



Fermi National Accelerator Laboratory  
Technical Division / Development & Test Dept.  
PO Box 500 MS 316  
Batavia, IL 60510  
FAX : 630-840-2383  
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# Niobium Heat Treatment Study Report

C. Chapman

**Abstract:** One of the fabrication steps for superconducting RF cavities made of bulk niobium is a high temperature heat treatment. Heating metal results in the positive effect of hydrogen outgassing as well as the uncertain effect of grain growth, which is known to lead to loss of strength. Polishing and lightly etching variously heat treated niobium samples reveals grain boundaries. This facilitates grain size measurements, which can be related to yield stress. Hydrogen partial pressures tracked during heat treatment allow outgassing comparisons. The grain size comparisons show a small amount of grain growth and corresponding slight loss of strength. Comparing the hydrogen partial pressures indicates more hydrogen depletion at 800C, five hours than at 600C, ten hours.

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## **Purpose**

To investigate various heat treatment recipes for niobium by measuring average grain size in order to calculate yield stress using a Hall-Petch plot. And to investigate heat treatment effects on hydrogen.

## **Introduction**

It is commonly known that heat causes grain growth. The merit of larger grains is to reduce the number of grain boundaries, which are potential emitters and sources of surface resistance. However, the drawback is loss of strength, which might lead to cavity deformation. Another factor to consider when heat treating is Q-disease, a lowering of quality factor caused by hydrogen in the niobium. As niobium is heated, hydrogen outgassing takes place, reducing and even eliminating Q-disease. These factors must all be taken into account when deciding how to heat treat the niobium that will be used to make accelerator cavities.

The goal of this study is to determine the best heat treatment taking into account yield strength reduction and hydrogen outgassing. Several steps will be undertaken to make this determination. First, niobium samples will be treated over a range of temperatures and durations, henceforward known as tests. The furnace temperature increases at a rate of 300C per hour, and the total pressure inside the furnace is less than  $10^{-7}$  Torr after pump down and remains below  $10^{-6}$  Torr throughout the heat treatment.

Table 1 shows a summary of the 3<sup>rd</sup> harmonic, batch 1, Wah-Chang niobium samples involved in the heat treatment tests. All the samples will undergo the degreasing process of an ultra-sonic detergent bath and subsequent ultra-pure water rinse. This will be followed by a 100 micron BCP etch using a mixture of one part hydrofluoric (HF), one part nitric (HNO<sub>3</sub>), and two parts phosphoric (H<sub>3</sub>PO<sub>4</sub>) before heat treatment and be stored in dry nitrogen until heating. The six control samples will go through this etch and storage as well, though they will not be heat treated. Additionally six more sample sticks will be cut from a sheet of niobium to facilitate practicing the polishing procedure; however, they will not have an initial 100 micron BCP etch. See Appendix A for a more specific sample history.

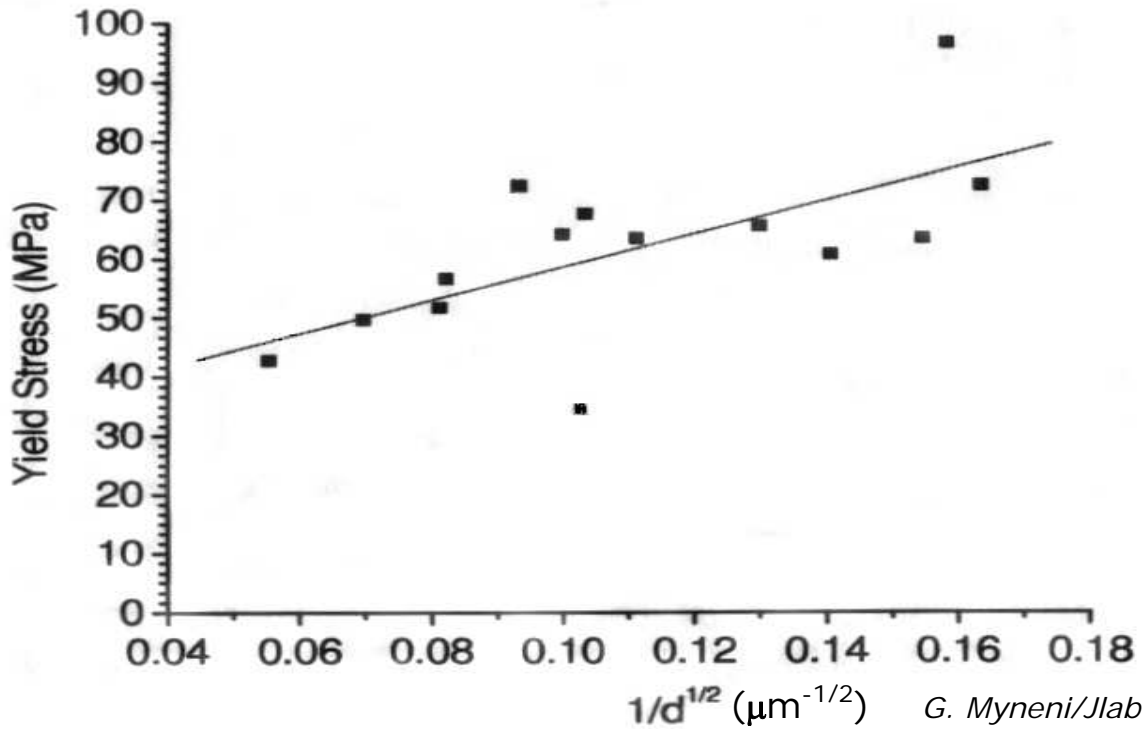
One sample from each furnace run and control set will be polished and slightly etched to bring out grain boundaries from which average grain size may be determined. Once the grain size is known, a Hall-Petch plot<sup>1</sup> (Figure 1) will be used to determine the yield stress of the sample. Data collected during the heat treatment will be used to examine hydrogen content. These methods and procedures are described in the various appendices.

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<sup>1</sup> G. R. Myneni, S. R. Agnew. "Elasto-Plastic Behavior of High RRR Niobium: Effects of Crystallographic Texture, Microstructure and Hydrogen Concentration." *1st International World Symposium in Materials and Vacuum Systems: November 11-12, 2002; AIP Conference Procedure 671*. Virginia: JLAB, 2003.

<b>Run 16</b>	Test 1	800C 5hr	4 Nb sticks, 2 magnetization samples
<b>Run 17</b>	Dry Run Clean-Up	800C 5hr	no samples
<b>Run 18</b>	Test 2	800C 2hr	4 Nb sticks
<b>Run 19</b>	Test 3	800C 1hr	4 Nb sticks
<b>Run 20</b>	Test 4	700C 5hr	4 Nb sticks
<b>Run 21</b>	Test 5, "Plateau"	500C 24hr, 800C 5hr	4 Nb sticks
<b>Run 22</b>	Test 6, "JLAB Recipe"	600C 10hr	4 Nb sticks
<b>Run 23</b>	Dry Run	600C 10hr	no samples
	Control	No Heat Treatment	6 Nb sticks
	Cut Samples	No Heat Treatment	6 Nb sticks

**Table 1:** Heat Treatment Study Sample List

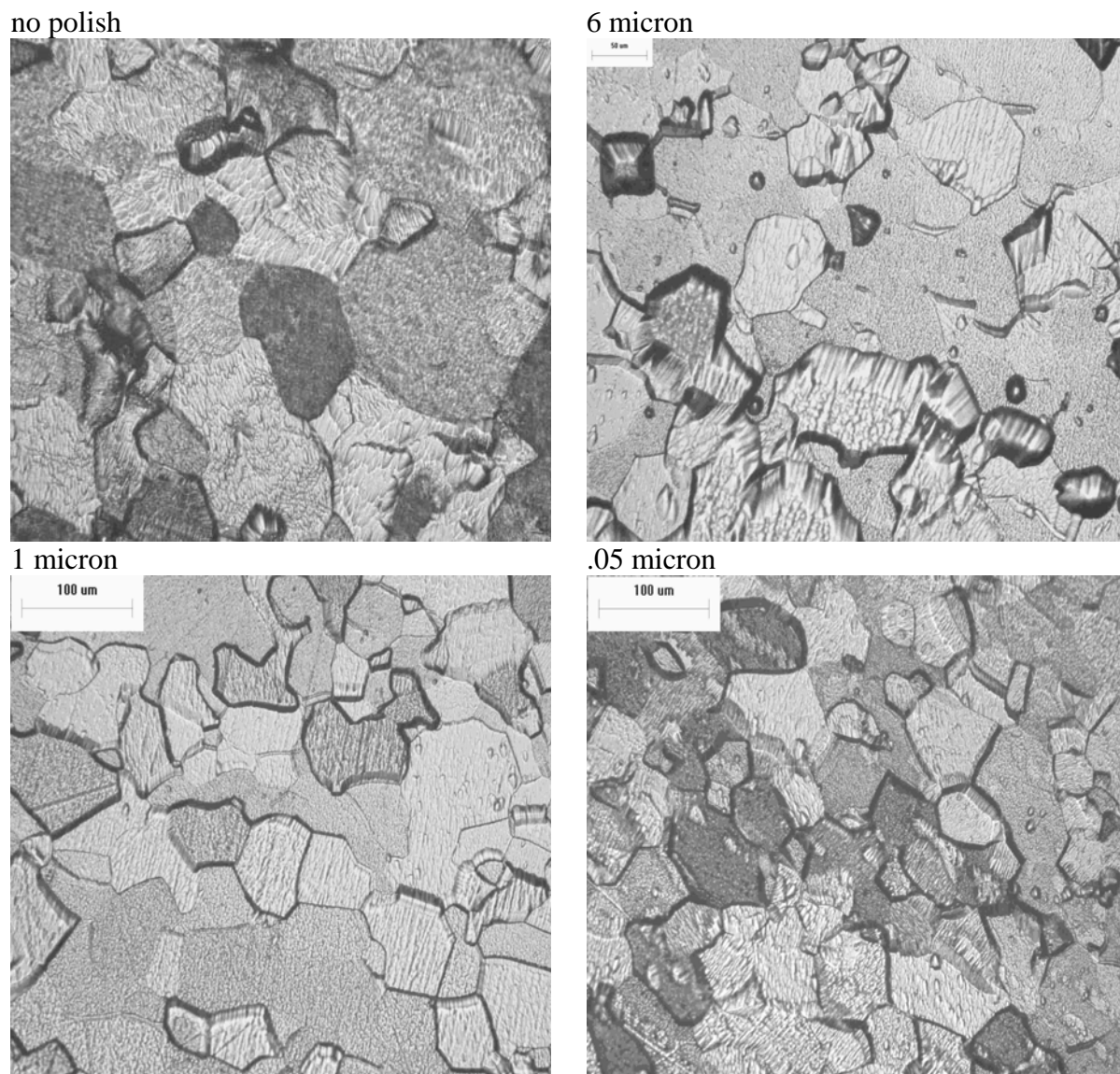


**Figure 1:** Hall Petch Plot for Wah-Chang Niobium

## **Results**

### ***Polishing***

Figure 2 shows images of a practice sample before BCP etching that has been polished to various levels. All the images were all taken at the same magnification. Appendix C gives more information on the polishing procedure. The images all look about the same. Therefore, it was initially determined that no polishing would be necessary. However, when a BCP etched sample was examined after only a short final etch, no grains could be seen. Consequently, some polishing would be necessary. A polish of 6 microns was deemed sufficient to see the samples' grains. The streaky places on the 6 micron image, as well as many other images shown elsewhere, are believed to be etching pits—residue left from the short acid etch performed under the microscope. The etching pits should not be a concern for the cavity production as they exist only after the polishing process.



**Figure 2:** Various Finish Polishes

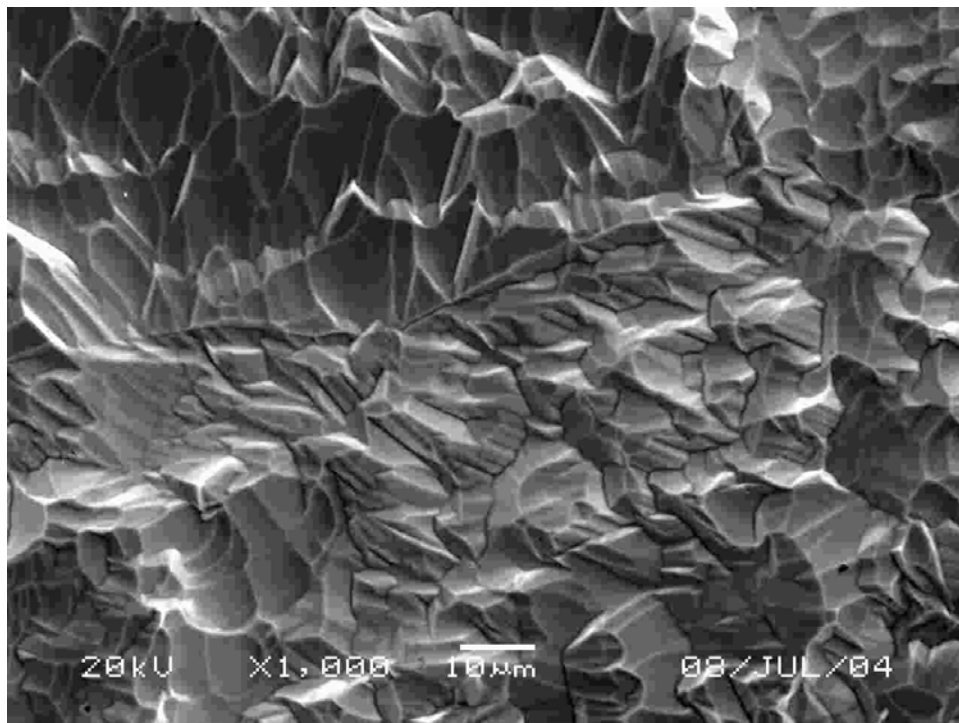
### ***Image Gallery***

The image gallery is composed of the best quality and most interesting images taken during the course of the heat treatment study. The captions give the image filenames. The last picture of each group was taken with the MicroStar optical microscope and all optical images have the same marker length though some do not have markers. The rest of the images were taken with the JEOL Scanning Electron Microscope (SEM). The filename is also under the image and some of the SEM pictures have additional information after the filename if it is not included on the image. This information is working distance, spot size, and beam type. SEI refers to the secondary electron beam, BEC refers to the composition feature of the backscattering beam, BET refers to the topological feature of the backscattering beam, and BES refers to the shadow feature of the backscattering beam. On most optical images the magnification is noted in the filename. See Appendix D for more information about the imaging software programs.

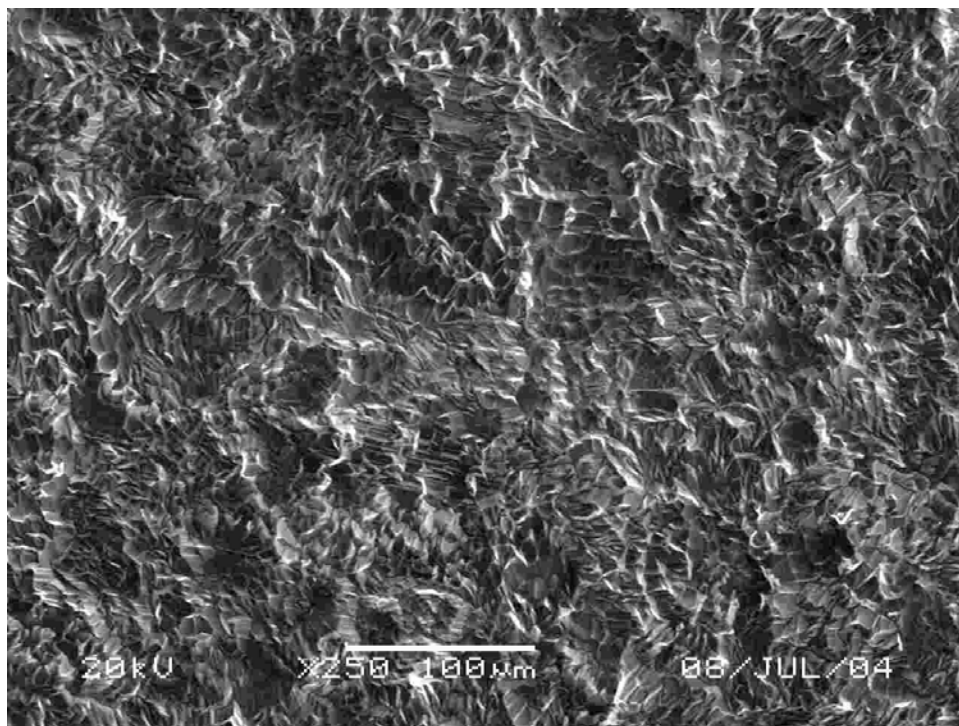
The rough appearance of the samples before polishing (Figures 3, 4, and 13) is addressed in the *Effects of BCP Etching* section following the *Image Gallery*. The surface roughness after BCP etching of the heat treatment study samples appear to be more pronounced than normal. The smooth image samples are all polished to six microns and lightly etched with an acid mixture of one part nitric ( $\text{HNO}_3$ ), one part hydrofluoric (HF), and two parts hydrochloric (HCl) acid. All images taken during the study are available on CD Rom.

A note of interest: the images all show a slight elongation in grains. This is probably due to the machining process used to produce the sheets of niobium from which the samples were cut.

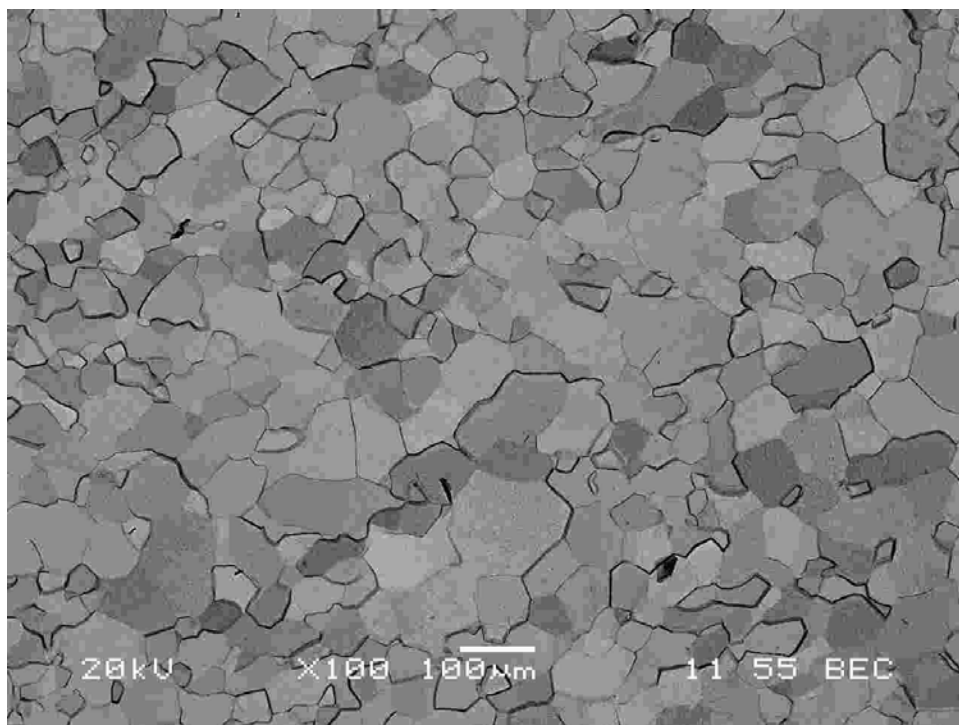
### ***Control Sample***



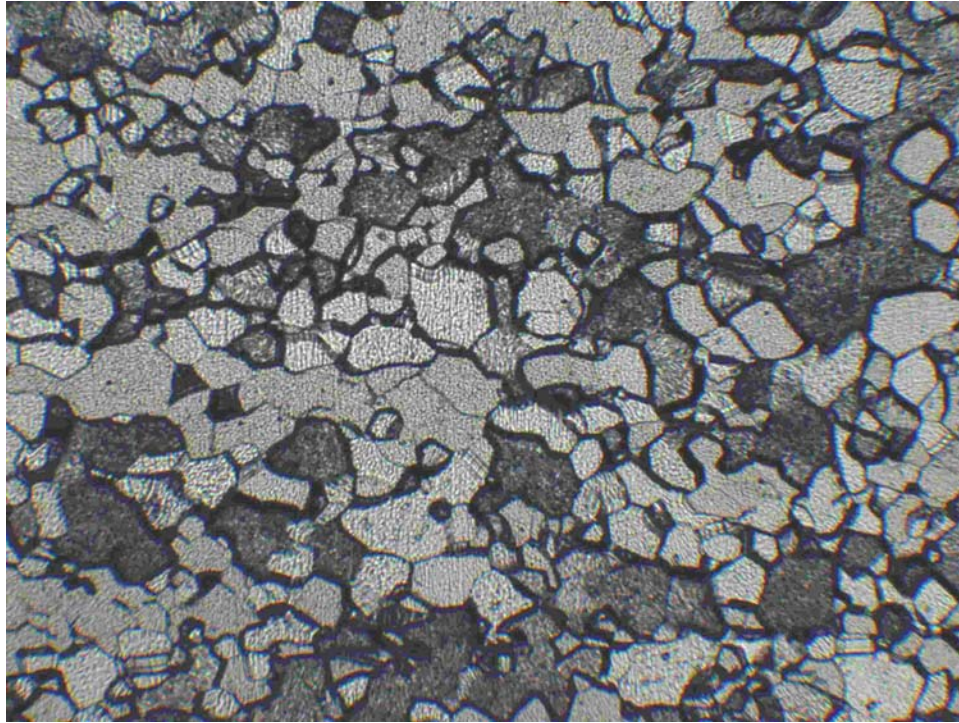
**Figure 3:** control 0708 clean 4: 11 65 SEI



**Figure 4:** control 0708 clean 6: 35 65 SEI



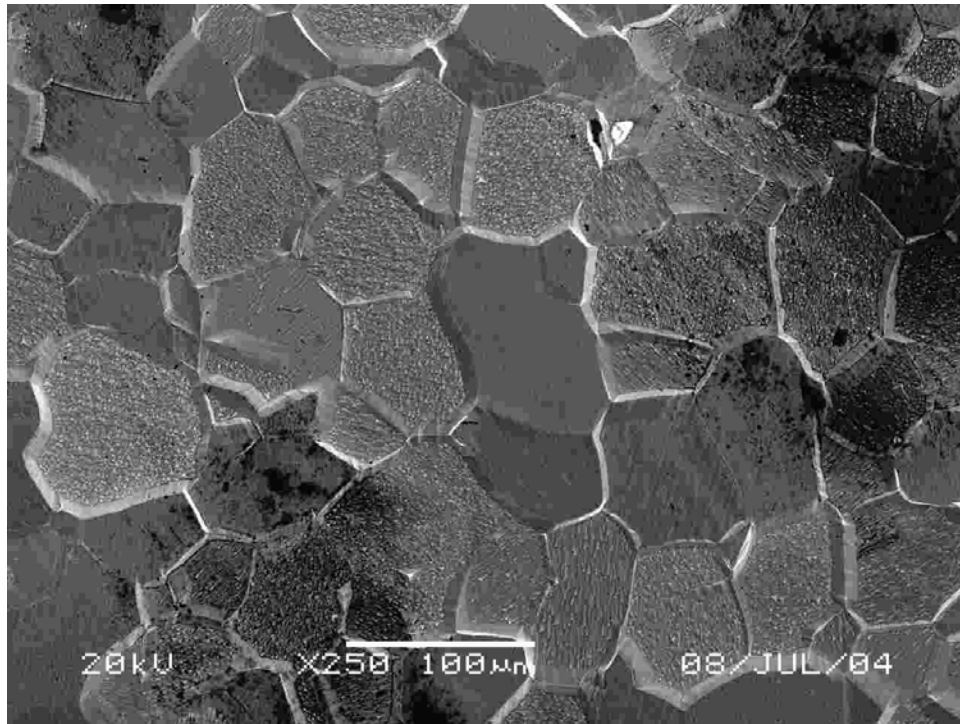
**Figure 5:** control 0712 comp2



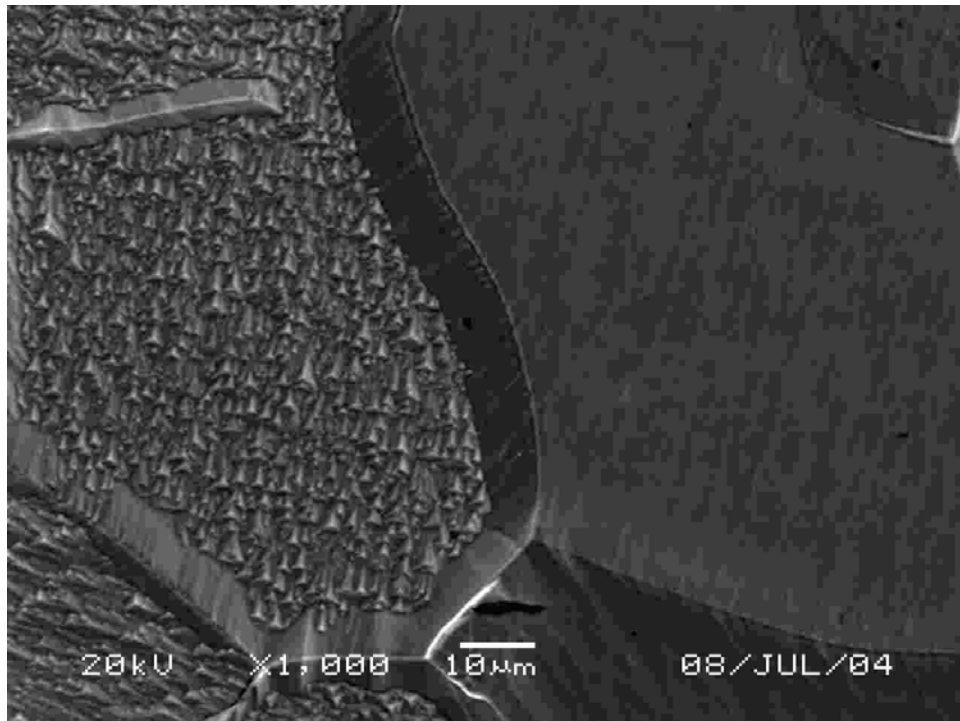
**Figure 6:** control 6um etch 6 5x 2

***800C 5 hour Sample***

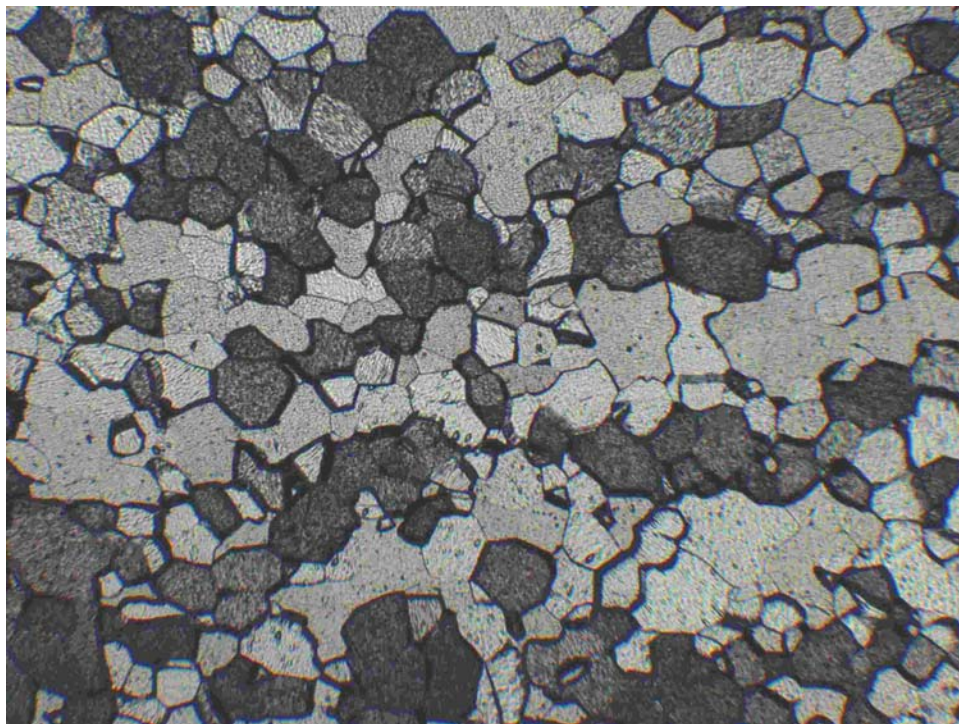
The varying appearance of the grain surfaces seen here and elsewhere is due to the polishing procedure. The smooth grains have retained the smoothness gained during the polish, while the rougher grains evidence etching pits from the light etch. Figure 8 is a magnified view of the center of Figure 7 and suggests even the smooth grains are not free of the etching pit effect. Figure 7 also clearly shows the step at the grain boundaries.



**Figure 7:** 800C 5hr 0708 2: 20 65 SEI

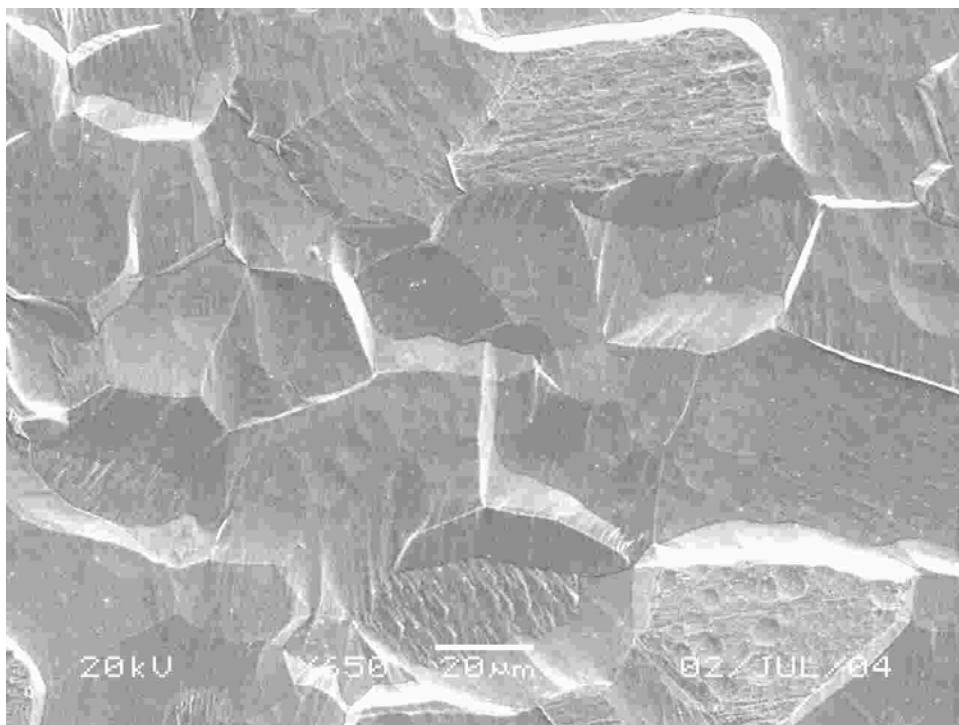


**Figure 8:** 800C 5hr 0708 3: 21 65 SEI

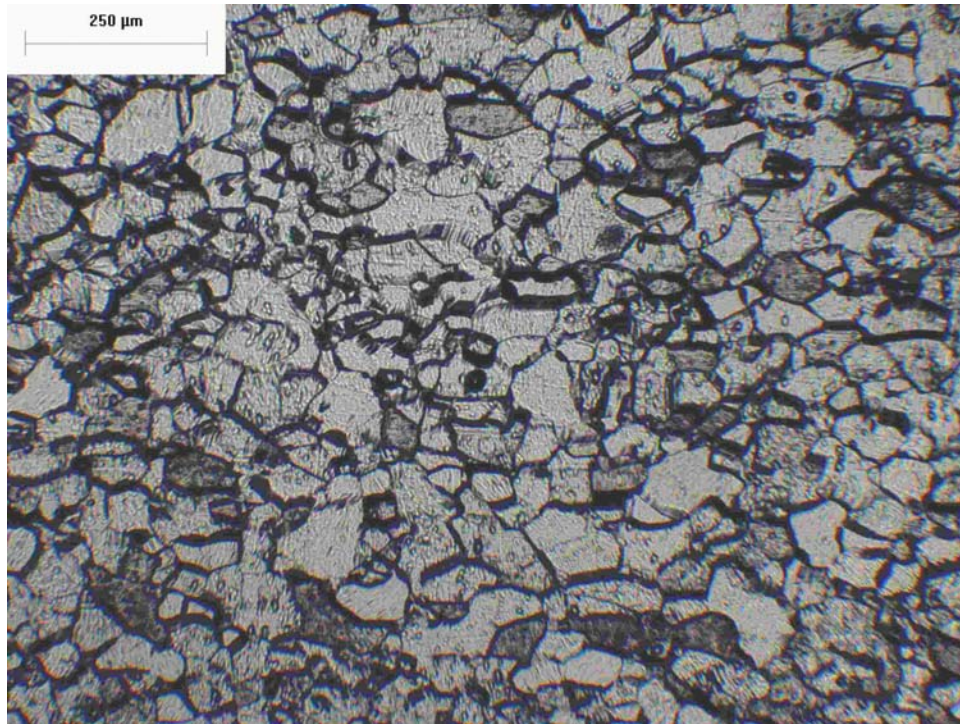


**Figure 9:** 800 5h 6um etch 6 5x 2

*800C 2 hour Sample*

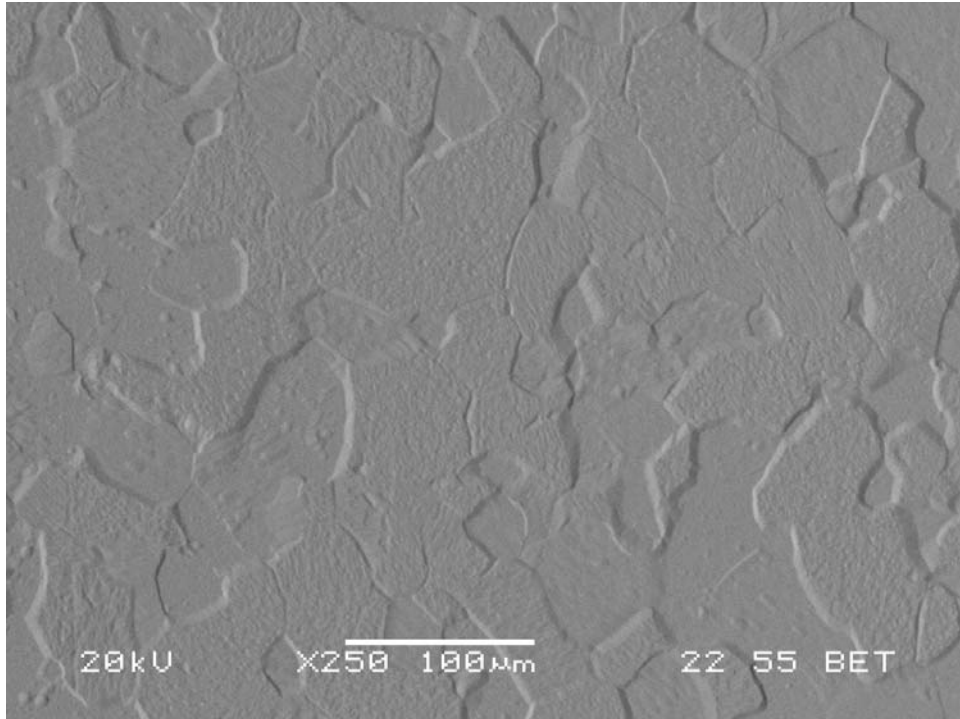


**Figure 10:** 800C 2hr 0702 4: 12 30 SEI

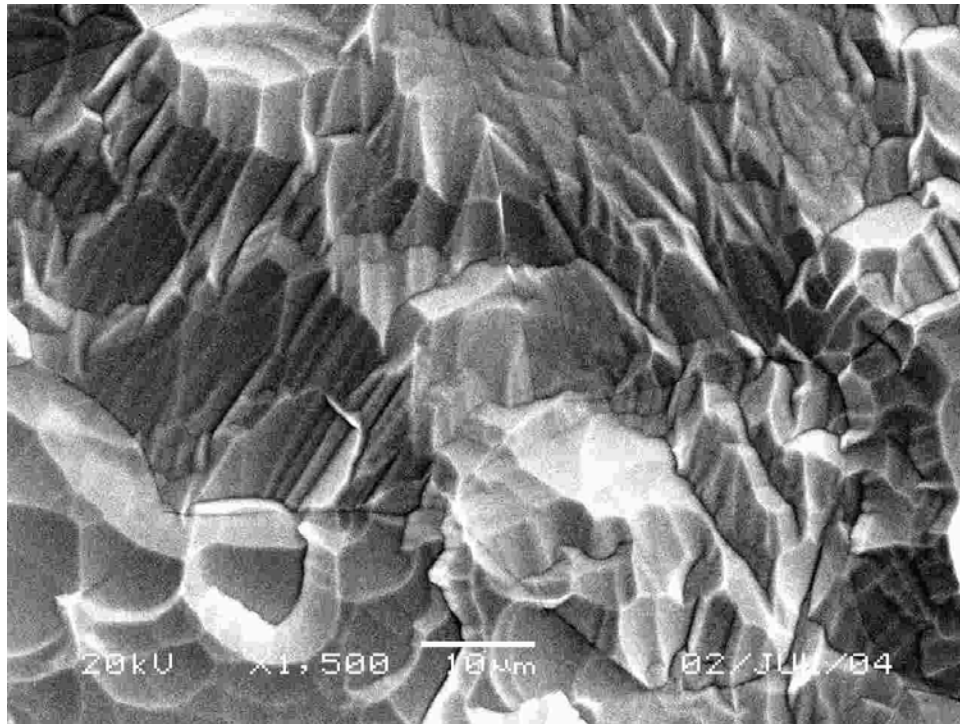


**Figure 11:** test 2 6um new etch 6 5x 1

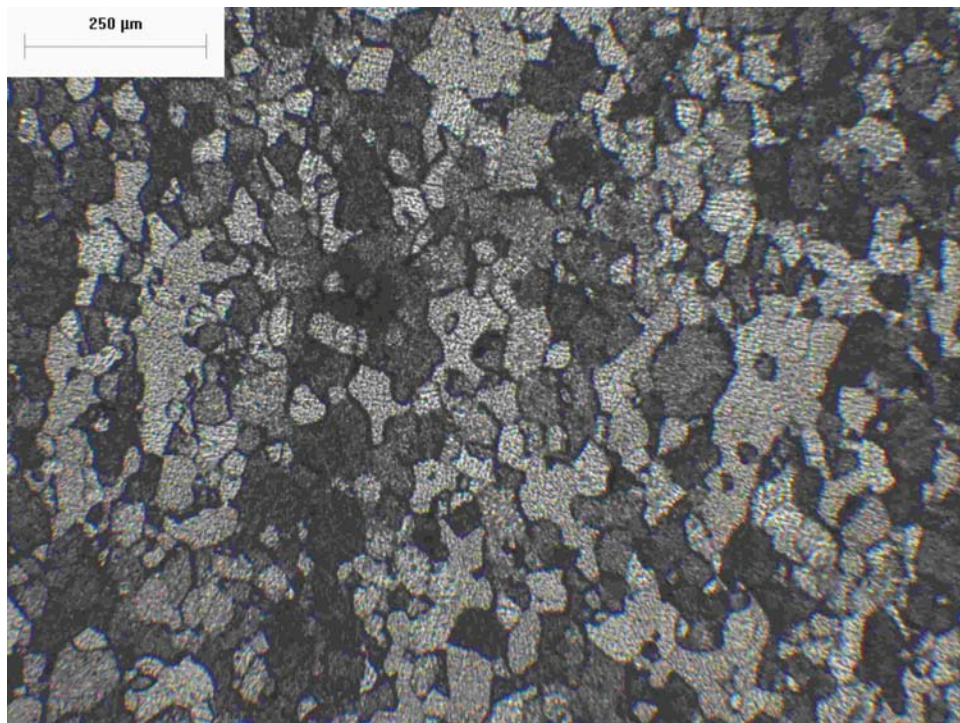
*800C 1 hour Sample*



**Figure 12:** 800C 1hr 0709 BEI

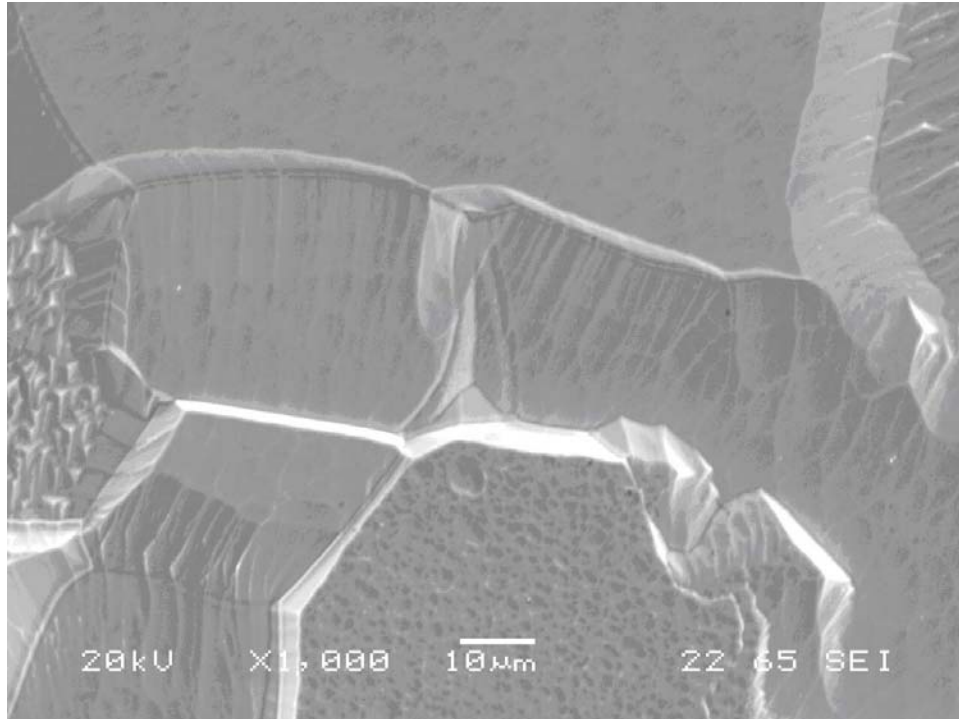


**Figure 13:** 800C 1hr 0702 clean: 12 31 SEI

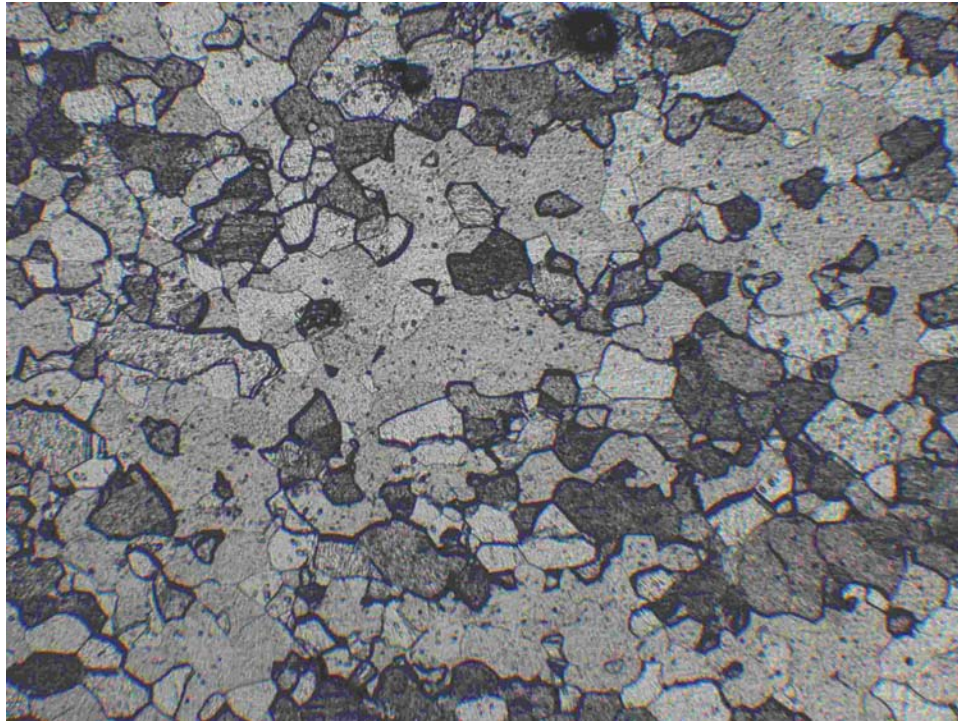


**Figure 14:** 800 1h 6um etch 6 5x 3

*700C 5 hour Sample*

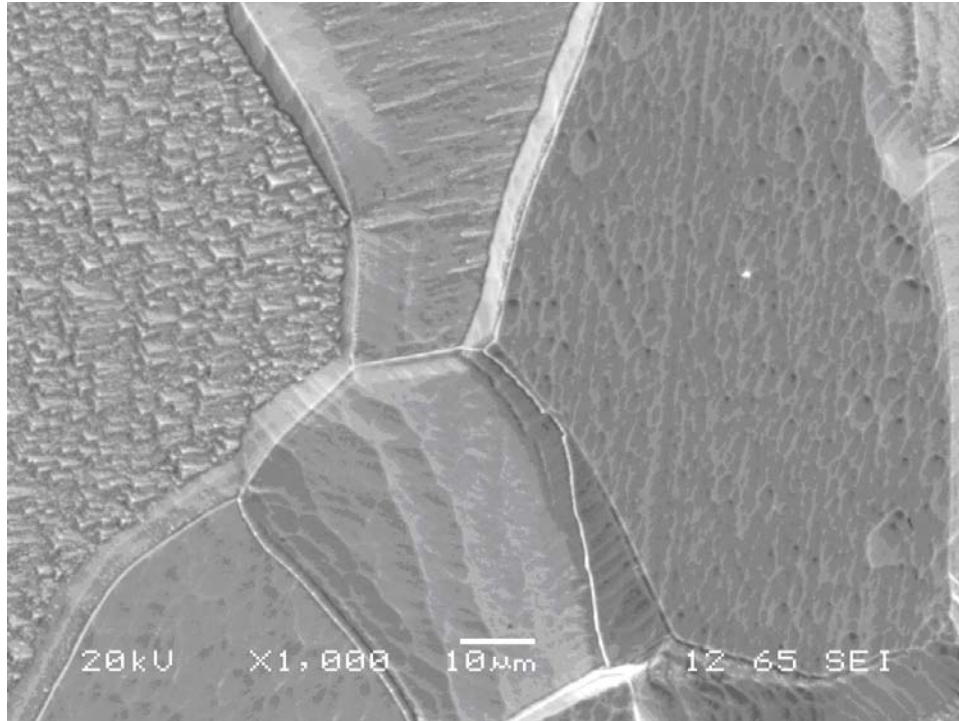


**Figure 15:** 700C 5hr 0709 3

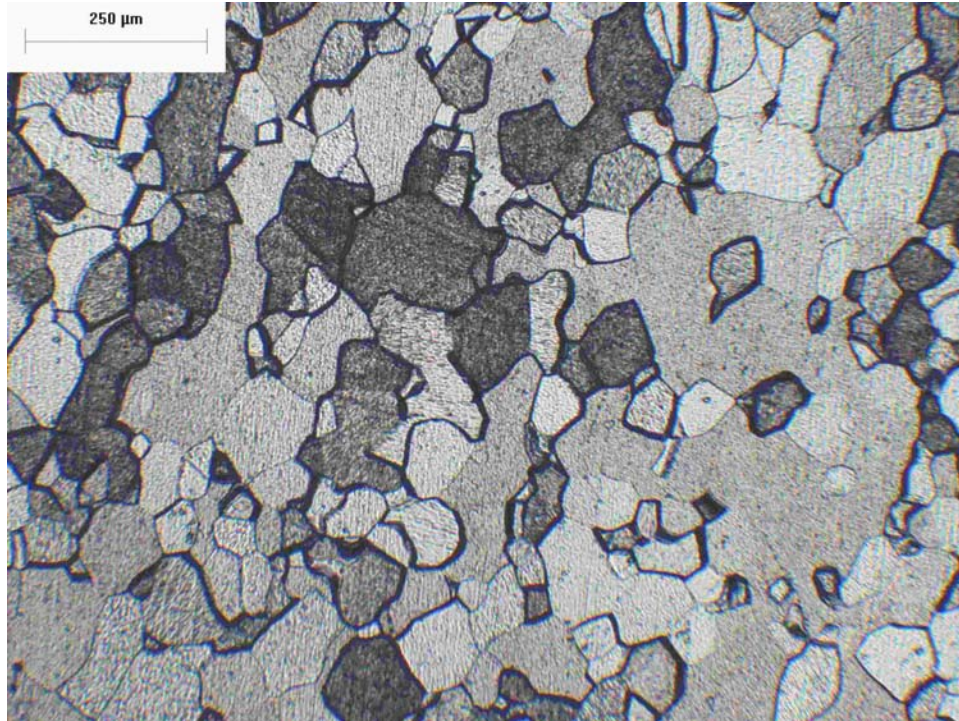


**Figure 16:** 700 5h 6um etch 6 5x 3

*Plateau Sample*

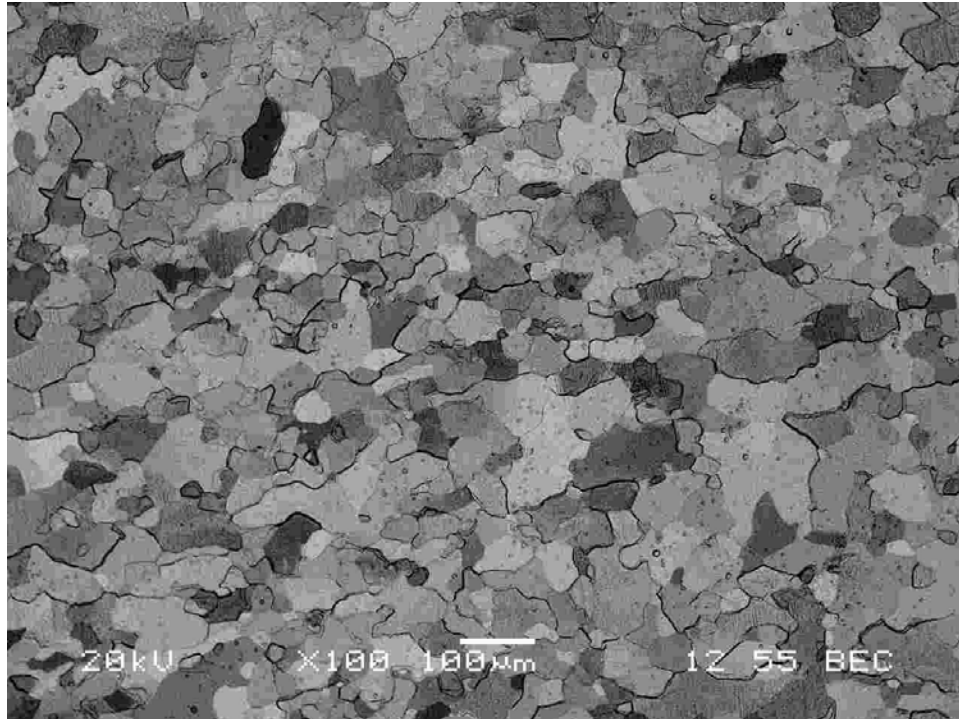


**Figure 17:** Plateau 0728 1000x

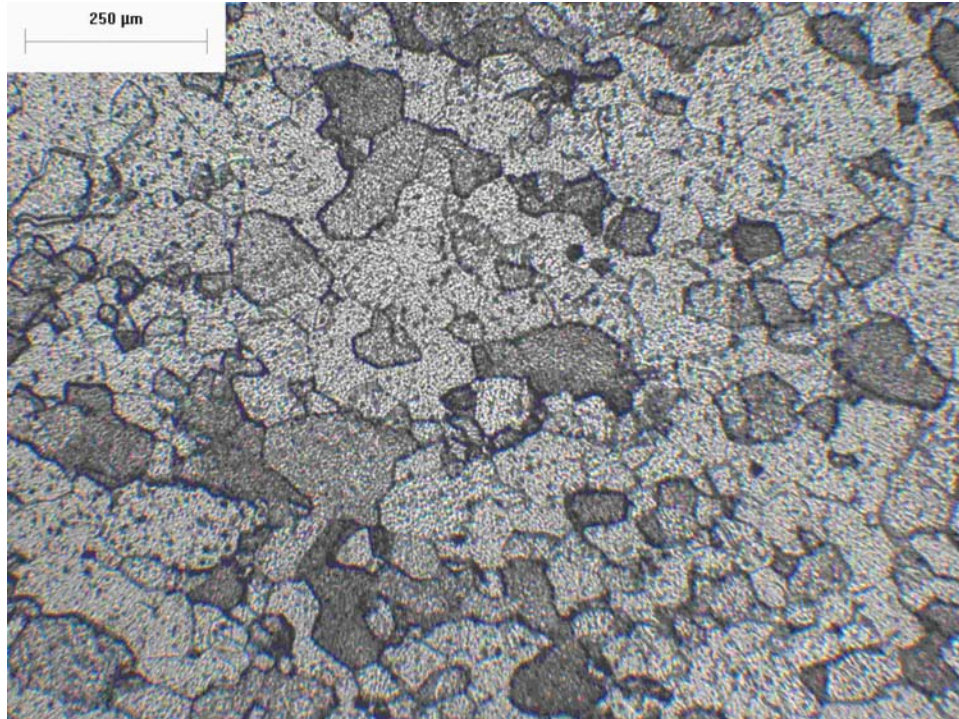


**Figure 18:** Plateau etched 65x 1

*600C 10 hour Sample*



**Figure 19:** 600C 10h 0802 comp2



**Figure 20:** 600 10h 6um etched 65x 1

Optical microscopes produce images sufficient to determine grain size; however, the scanning electron microscope yields clearer images with better magnification capabilities. An image of the control sample using the backscattering composition function was the easiest image in which to count grain boundary intersections. If samples are etched sufficiently, this function will yield images best suited for grain measurements. Traditional secondary electron scattering images are preferred for publication images as the working distance can be at its shortest and the spot size can be larger, both of which yield better resolution.

The images taken throughout the course of the study all show a slight grain elongation in the direction of rolling, etching pit effects, and stepped grains boundaries. These steps have previously been determined to be approximately one micron in height.

### ***Effects of BCP Etching***

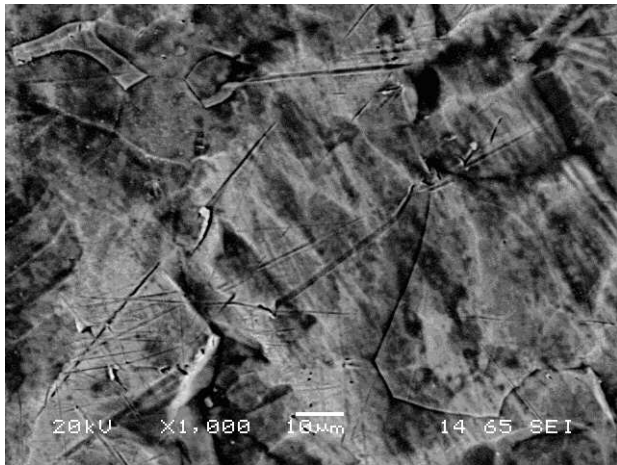
An image of the surface of an unpolished control sample spurred some concern about the surface roughness of BCP etched samples, and further investigation ensued. Additional samples possessed the same rough appearance as the control sample. Though each sample was not examined, it is believed they all share this unexpected ruggedness. Other labs typically see a smoother surface<sup>2</sup>. A note of interest is that the cut surfaces appear slightly rougher than the uncut surfaces of the samples, though the 100 micron BCP etch should remove most artifacts of the cutting process. The samples for the heat treatment had been placed in individual test tubes filled with the BCP acid mixture. The temperature of this mixture was regulated to a range between 5 and 15C, and the samples were agitated periodically throughout the 100 minute etch. Each minute of etching typically removes a surface layer of one micron depth.

The first step in the investigation was to determine if the temperature of the acid mix caused the extra roughness of the sample surfaces. It was found that keeping the acid mixture between 5 and 15C did not result in smoother surfaces than allowing the acid temperature to climb unimpeded to reach 26C. The second line of Figure 21 shows these surfaces, both of which look like the typically seen surfaces. However, the new samples were etched in beakers of acid rather than test tubes, so there was much more acid per surface area for the new, smoother surfaces. Also considered was the effect of the second, 'light' etch of twenty minutes after heat treatment in addition to the original 100 micron etch. This smoothed the original, rough surface to the more expected result, but, again, this etch was performed in the larger amount of acid. The second image of the first line of Figure 21 shows the initial rough surface, and the first image of the third line shows the smoother surface that resulted after the second BCP etch.

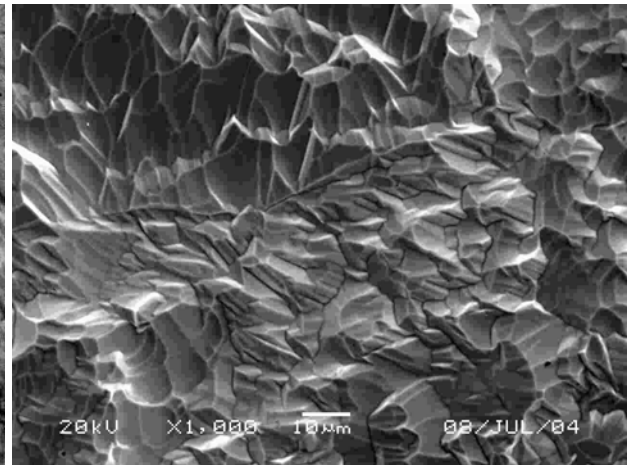
A final test of etching a newly cut sample in a test tube of acid was performed, which produced surfaces similar to the larger amount of acid (second image, third line). The only factors left unchecked are the amount of agitation of the acid during the etching process and the exact acid mixtures used. A note of interest is that the cut surfaces appear slightly rougher than the still rough, uncut surfaces of the samples. Perhaps the extremely rough surfaces from the original heat treatment samples are a result of stagnant acid or a slightly different acid composition, or perhaps they are simply an anomaly. The first image in Figure 21 shows a cut, unpolished, unetched sample.

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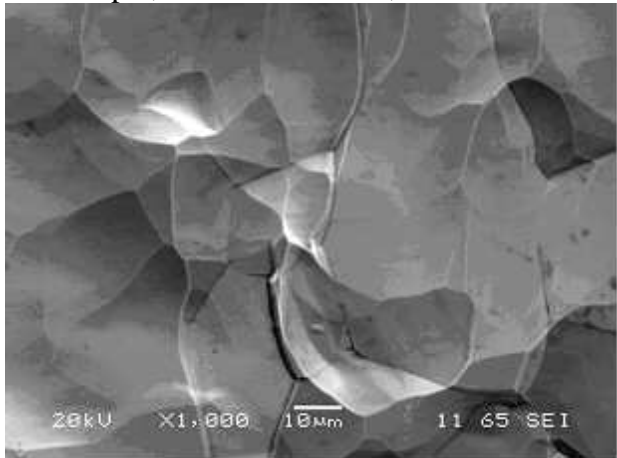
<sup>2</sup> X. Singer, "High Purity Niobium for Tesla Test Facility," *Proceedings of the Tenth UHPM, 2003, Matériaux et Techniques* 7-8-9 2003.



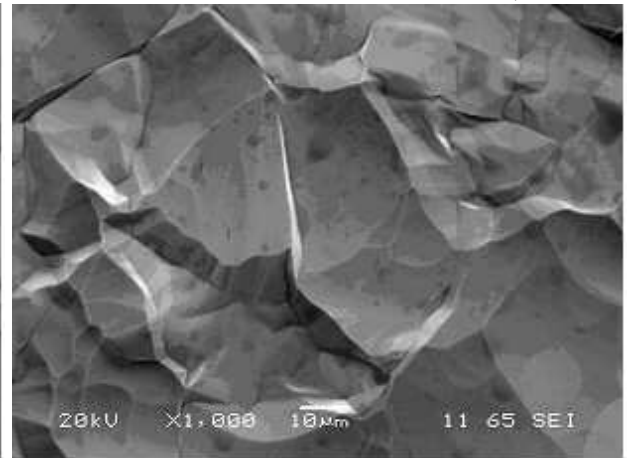
Cut sample, no heat treatment, no BCP etch



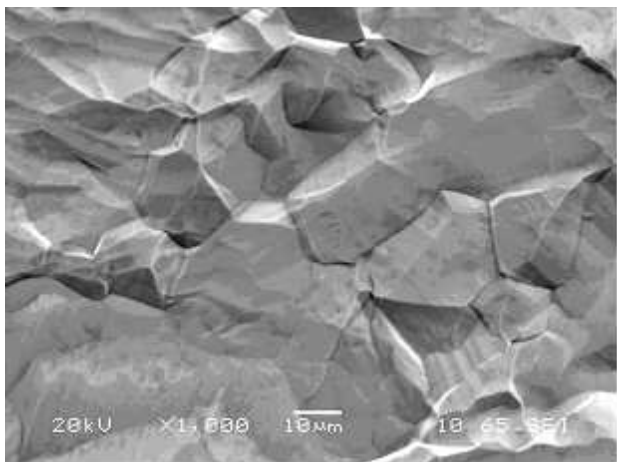
100µm BCP etch, control



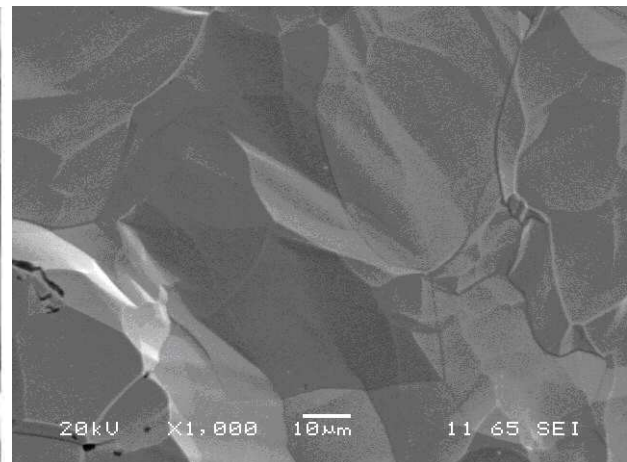
100µm BCP etch 21-26C



100µm BCP etch 10-12C



100µm BCP etch, 700C 5hr, 20µm BCP etch 10-12C



100µm BCP etch, less acid, 6-12C

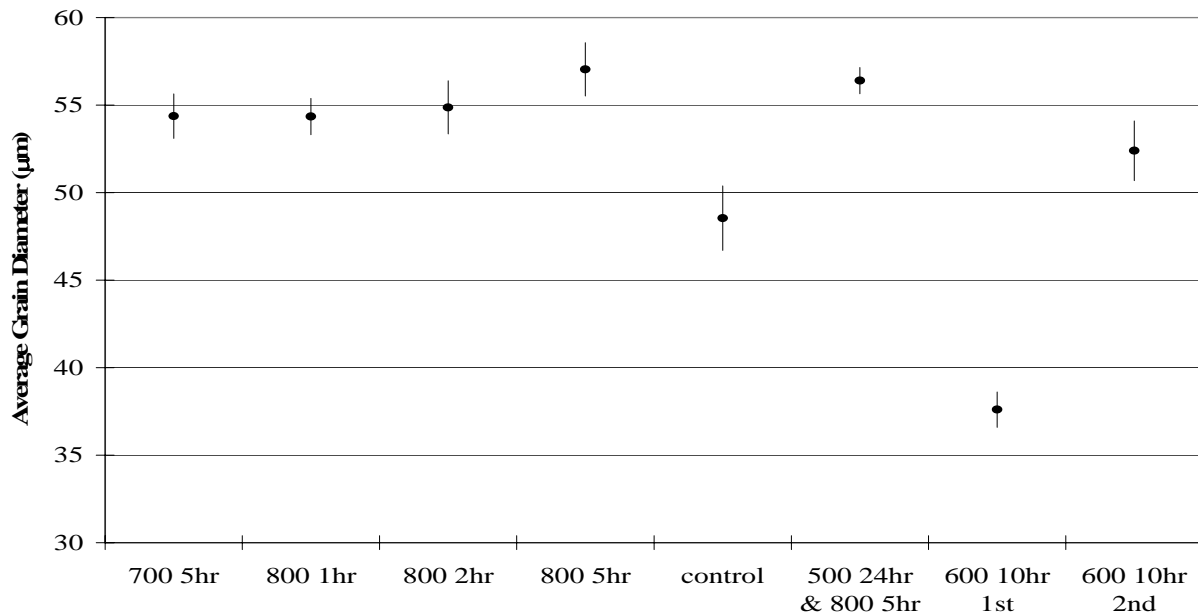
**Figure 21: Surface Roughness Comparison**

### Grain Size Measurements and Yield Stress

Table 2 gives the results of the grain size measurements, which were made according to the ASTM E112 procedure (Appendix E). The values in the table include the average grain diameter, accompanying statistical data (Appendix E), and ASTM grain size for the control and variously heat treated samples. ASTM grain size for niobium should be 6 or finer and is typically around 4 or 5. Figure 22 gives a graphical representation of the samples' average grain diameters along with the standard deviations. Figure 23 shows the grain diameters and yield stresses for the samples normalized to the control sample. Figure 24 shows a comparison of the grain diameters from this study and a previous study performed at JLAB<sup>3</sup> for treatments of 600C for 10 hours and 700C, 750C, and 800C for six hours.

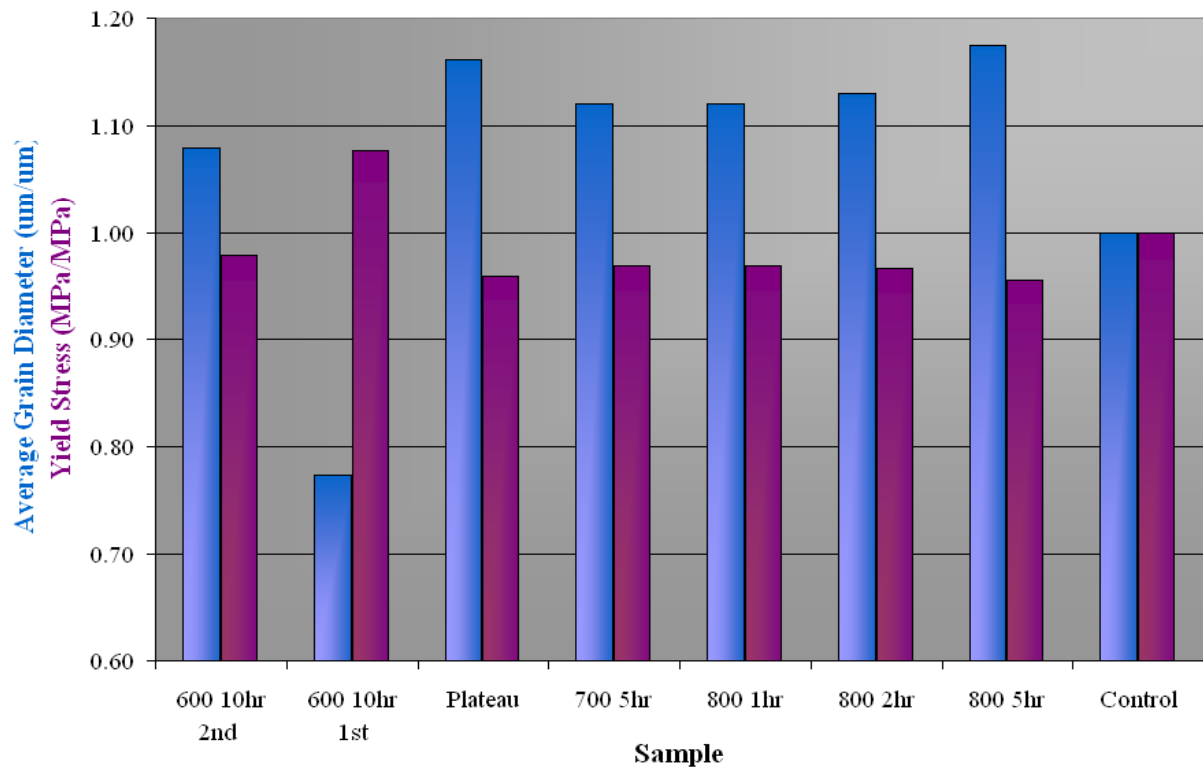
Sample	Grain Diameter (μm)	Uncertainty in Grain Diameter	ASTM Grain Size, G	Standard Deviation, s (μm)	Confidence Interval, 95% CI (μm)	Relative Accuracy, %RA
600 10hr 2 <sup>nd</sup>	52.39	1.443	5.22	5.89	3.74	7.15
600 10hr 1 <sup>st</sup>	37.58	1.495	6.18	3.53	2.24	5.97
Plateau	56.38	1.217	5.01	2.59	1.65	2.92
700 5hr	54.37	1.239	5.11	4.40	2.80	5.15
800 1hr	54.35	1.236	5.12	3.58	2.27	4.18
800 2hr	54.87	1.242	5.09	5.26	3.34	6.10
800 5hr	57.04	1.239	4.98	5.28	3.35	5.88
Control	48.54	1.239	5.44	6.39	4.06	8.37

**Table 2:** Average Grain Diameter and Statistical Analysis

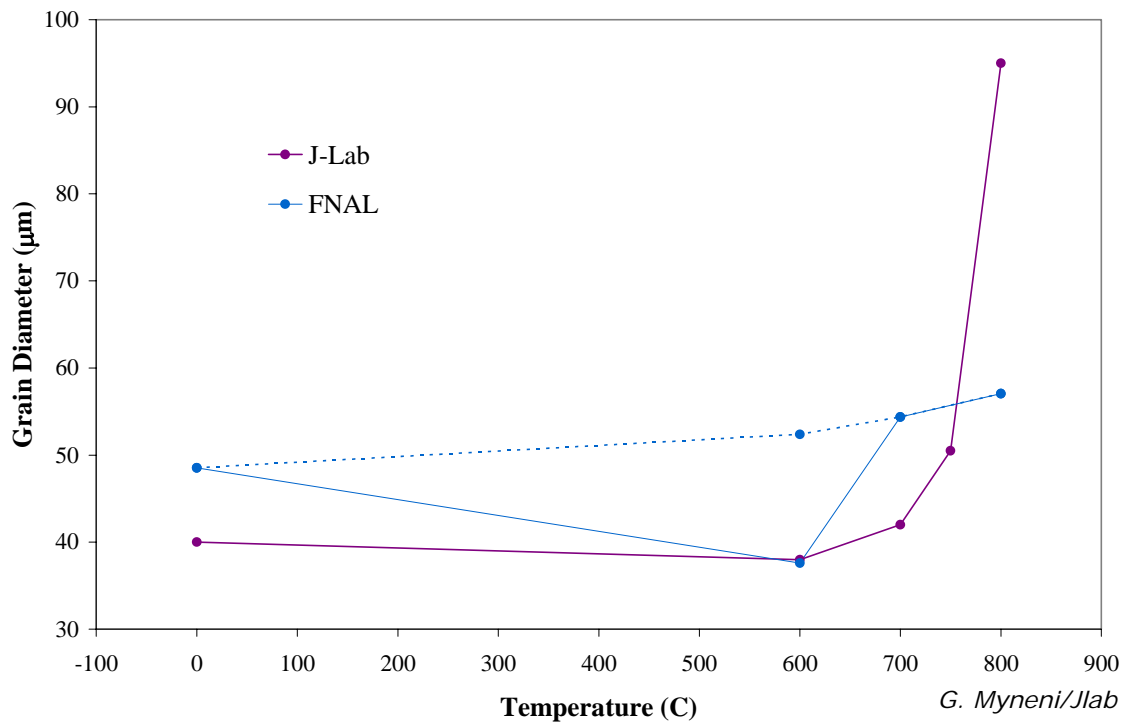


**Figure 22:** Grain Diameter with Standard Deviation

<sup>3</sup> G. R. Myneni, S. R. Agnew. "Elasto-Plastic Behavior of High RRR Niobium: Effects of Crystallographic Texture, Microstructure and Hydrogen Concentration." *1st International World Symposium in Materials and Vacuum Systems: November 11-12, 2002; AIP Conference Procedure 671*. Virginia: JLAB, 2003.



**Figure 23:** Average Grain Diameter and Yield Stress Normalized to Control Sample



**Figure 24:** FNAL and JLAB<sup>3</sup> Grain Size Study Comparisons

Table 3 shows the yield stresses calculated from the Hall Petch plot in Figure 1, as well as the allowable loads at the iris of a 3<sup>rd</sup> harmonic cavity with inner radius of 15mm, and thickness of 2.2mm. Appendix F explains the procedures for obtaining these values.

Sample	Yield Stress (MPa)	Allowable Load at Iris	
		kN	lb <sub>force</sub>
600 10hr 2 <sup>nd</sup>	69.68	7.75	1743
600 10hr 1 <sup>st</sup>	76.68	8.53	1918
Plateau	68.29	7.60	1708
700 5hr	68.97	7.68	1725
800 1hr	68.98	7.68	1726
800 2hr	68.80	7.66	1721
800 5hr	68.07	7.57	1703
Control	71.19	7.92	1781

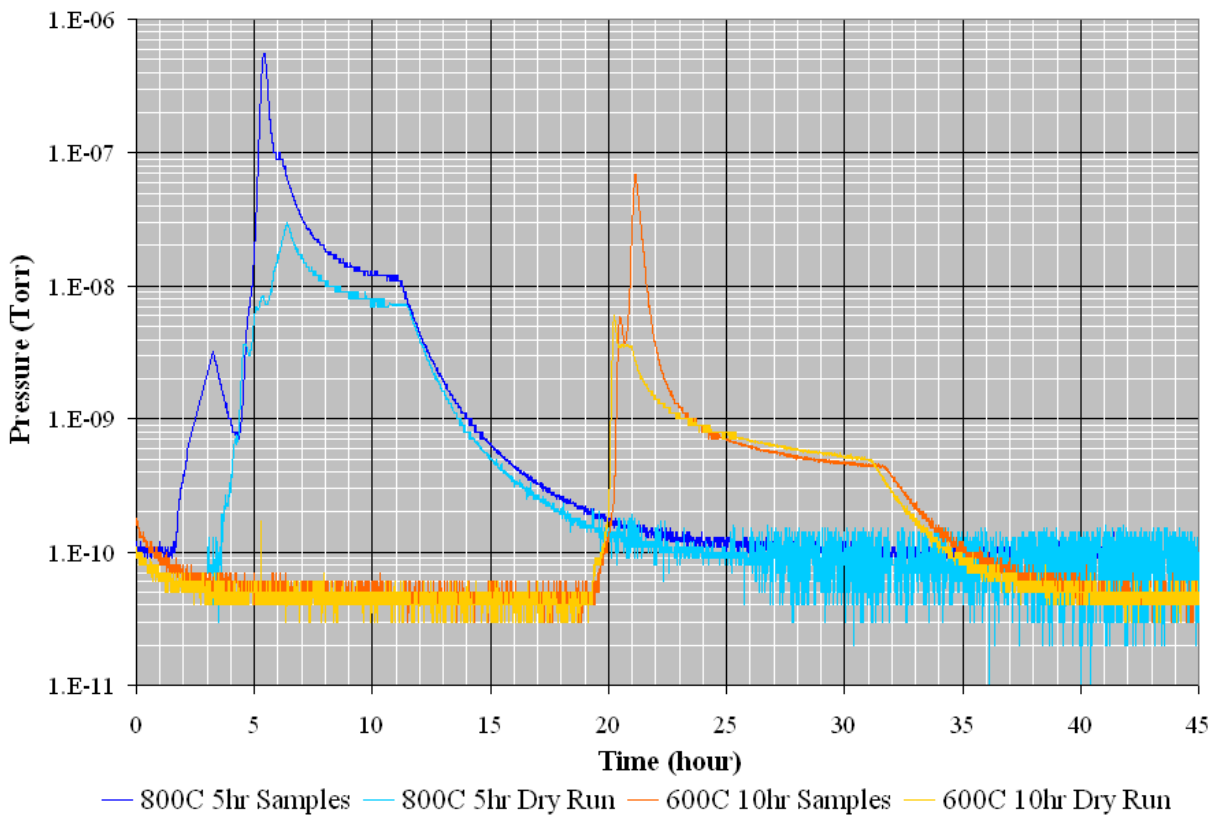
**Table 3:** Yield Stress and Allowable Load

All but one of the measured ASTM grain sizes fall within the expected range of being less than 6. The measurement of the first 600C, 10 hour sample resulted in smaller grains than the control sample and a larger ASTM grain size than expected, which prompted a second measurement. The largest difference in grain diameter, excluding the odd 600C point was between the control sample and the 800C, five hour sample; this was a variation of slightly more than 15%. The small grains for the 600C heat treatment were also found at JLAB. However, the large grains at a heat treatment temperature of 800C seen at JLAB were not seen here. The JLAB study noticed more grain size non-uniformity in the Wah-Chang niobium than in other sources of niobium. The grain diameter variation within a single heat treated sample was larger than the grain diameter difference between the heat treatments. The control sample diameter did fall outside the standard deviation range for any heat treated sample. The change in grain diameter between the control sample and the 800C, five hour sample resulted in only a slight loss of strength. The yield stress has little variance between the various heat treatments, and thus the allowable load changes only slightly.

### **Hydrogen**

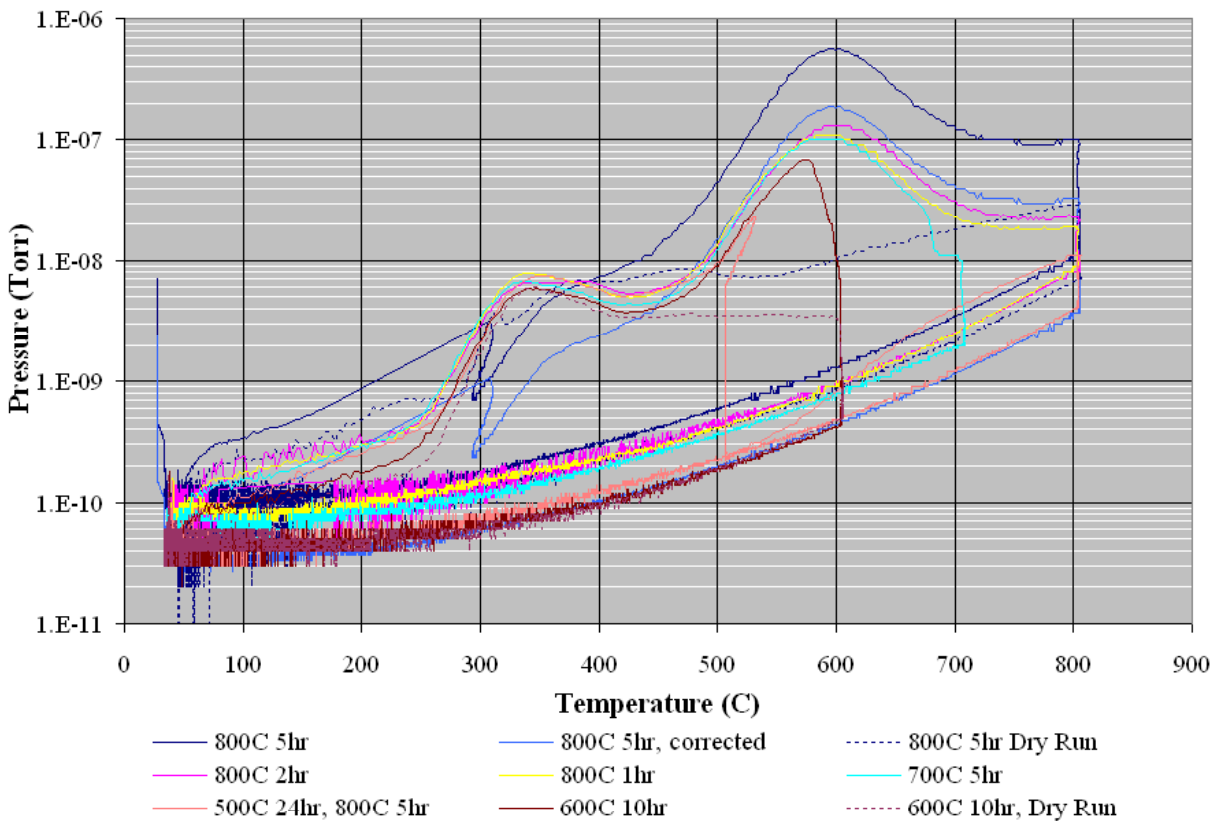
As mentioned in Appendix B, the partial pressures of various gases are tracked during vacuum heat treatments. Hydrogen (H<sub>2</sub>) is of particular interest due to its effect on quality factor. According to Anne-Marie Valente of JLAB, when this data is plotted against temperature, pressure peaks should be noticeable at particular temperatures, typically around 350C and 580C. For the thermal desorption spectroscopy studies these results originate from, there is also a peak around 720C that is probably a result of the cooling process after heat treatment, and thus should not be visible in this study. The peak at 350C is from a surface effect, while the other peak evidences trapped hydrogen molecules escaping from the samples. However, it could be possible that the much larger surface area of the furnace chamber and shelving is where this phenomenon comes from rather than the small sample surfaces. Dry runs—no samples on the shelving unit in the furnace chamber—were conducted at 800C, five hours and at 600C, ten hours in the hopes of seeing a difference in hydrogen partial pressures that would indicate hydrogen escaping from the heat treatment samples.

A comparison of the runs with empty shelving to similar runs with samples (800C, five hours and 600C, ten hours) indicate a noticeable amount of hydrogen is escaping from the samples (Figure 25). The extra peak in the 800C, five hour run with samples is due to a power outage that interrupted the temperate ramp around 300C.



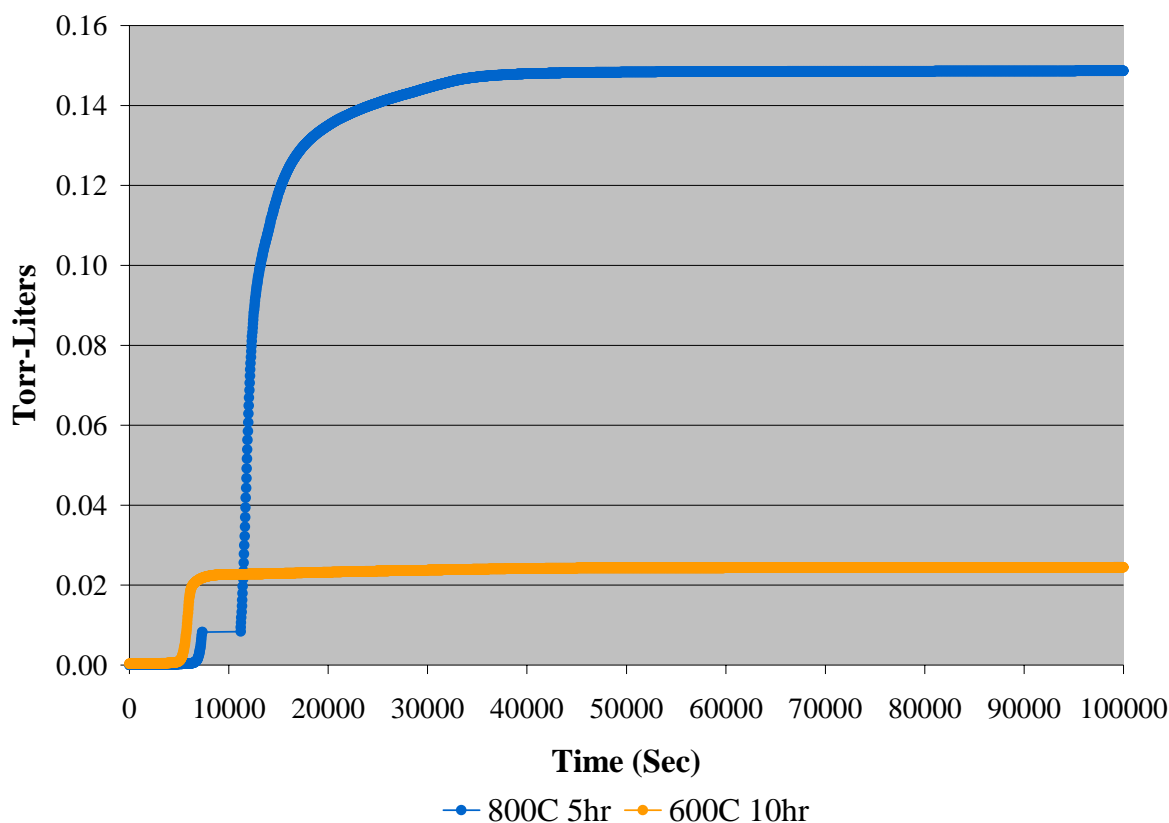
**Figure 25:** Empty Shelving Runs Compared to Sample Runs

Examination of the partial pressure versus temperature plots shows the expected peaks at 350C and 600C for most of the samples (Figure 26). The Plateau run shows the first peak, but not the second, which would suggest that either the twenty four hours at 500C depleted the hydrogen. The 600C, ten hour run shows both peaks, but the second peak is shortened; it is not clear whether the long bake time releases as much hydrogen as the peaks at higher temperatures. The 800C, five hour run contained extra niobium in the form of two magnetization samples which tripled the surface area of the samples. This would account for the higher partial pressures. A line corrected for the extra surface area is included.



**Figure 26:** Hydrogen Pressure versus Temperature

Figure 27 shows a comparison of the outgassed hydrogen for the 800C, five hour and 600C, ten hour runs. To obtain the plots, the difference in hydrogen partial pressure for each temperature's dry and sample runs was calculated and integrated over time. The 800C data has been corrected for the extra sample area. After the integration, the pressures were multiplied by the ideal pumping speed for hydrogen by the furnace turbo-molecular pump, 480 L/sec. The actual pumping speed will be less than ideal due to baffling at the bottom of the furnace chamber and a ninety degree turn between this and the pump.



**Figure 27:** Comparison of Integrated Hydrogen Pressure Times Ideal Pumping Speed

The difference in hydrogen partial pressure between furnace runs with and without samples indicates a significant amount of hydrogen is escaping from the niobium samples during heat treatment. Thermal desorption spectroscopy measurements at JLAB show samples with a beginning hydrogen content of 200ppm retain 20ppm during a 600C, ten hour heat treatment and 10ppm during an 800C, five hour heat treatment. The integration of hydrogen in this study shows a larger amount of hydrogen escaping from the 800C treatment as the JLAB findings suggest, though this larger amount is greater than expected.

## Conclusions

After the polishing process, all the samples exhibit several features. The final, light etch causes pits to appear on many of the grains. The pits are unavoidable because a less aggressive acid mixture does not yield the same clarity of grain structure. Another common feature is a

noticeable step at the grain boundaries, which helps distinguish the grains from one another. The initial BCP etching affects surface smoothness. One stick of niobium does not greatly affect the temperature of the acid mixture, and thus temperature restriction is of small importance. However, larger surface areas could cause larger temperature increases, which are commonly held to be a problem during the etching process. The amount of acid used to etch the samples also appears to have little effect on surface roughness. At this point, the cause of the rough surface seen on the original samples before polishing remains unknown.

The relative accuracies of the grain size measurements are within the accepted limit of less than ten percent. The grains do grow when heat treated. The control sample grains had an average diameter of 48.5 microns, the 800C, five hour sample grains had an average diameter of 57 microns, and the 600C, ten hour samples had grains of 52.4 and 37.6 microns. The 37.6 micron measurement is unusual, but not unheard of, especially for Wah-Chang material, which is known to have odd variations in grain size. The grain growth results in no more than five percent loss of yield stress. The control yield stress is 71.2 MPa, the 800C, five hour yield stress is 68.1 MPa, and the 600C, ten hour yield stresses are 69.7 and 76.7 MPa. The increase of yield stress for the anomalous 600C sample is just above seven percent. The differences between the 800C, five hour and 600C, ten hour runs are twelve and two percent. It seems a carefully monitored heat treatment should not result in enough loss of strength for a cavity to deform under its own weight.

It should be noted that the Hall Petch plot used to calculate the yield stresses probably does not correlate exactly with the samples used in this study. The actual values of the yield stresses calculated here are probably a bit incorrect, but unless the actual yield stresses are vastly different, the comparisons are sufficient to show only a small variation in yield stress between heat treatments. More accurate results would come from direct measurement of yield strength through tensile testing. However, cryogenic facilities are not currently available on the testing machine and the improvement in values might not be enough to merit such a study.

In comparing the 800C, five hour and 600C, ten hour runs with and without samples, an increase in hydrogen partial pressure of a factor of ten can be seen when samples are in the furnace. Comparing the hydrogen pressure versus temperature for each of the runs indicates a heat treatment of 800C for five hours will result in the largest depletion of hydrogen from the material. Further tests of higher heat treatments might reveal even better hydrogen depletion, but such tests are outside the scope of this study. The pressure versus temperature plot shows the expected peaks around 350C and 580C, except for the Plateau run. The plateau at 500C for twenty four hours probably depletes the same amount of hydrogen as the second peak, but this is not known for certain. There is evidence the prolonged plateau did not significantly increase grain size. It is probably allowable to state that the plateau is not a detrimental effect of in the heat treatment process. The integrated hydrogen pressure for the 800C, five hour run is nearly seven times larger than the same for the 600C, ten hour run. Studies at JLAB do indicate a larger depletion of hydrogen for the 800C treatment, though not as great as seen here.

In light of the small change in grain size and yield stress, there seems to be no reason to change the heat treatment of niobium from 800C, five hours to 600C, ten hours, especially since the higher temperature appears to deplete more hydrogen.

## **Appendix A: Sample History**

All Nb sticks, excluding the Cut Samples, went through the same original process: they were cut from niobium sheets, degreased, underwent a 100 micron BCP etch, and were stored in test tubes in a dry nitrogen box in Lab 7 until heat treatment. After heat treatment, the sticks were placed in new test tubes and returned to the nitrogen box to await polishing.

### **Run #16**

Test #1

800C 5hr

4 Nb sticks, 2 magnetization samples

Magnetization samples given to Cris Boffo.

1 stick [800C 5hr] placed in N<sub>2</sub> filled test tube and given to Pierre, later given to Colleen to sit in desk drawer forgotten until 6-24.

6-24: 800C 5hr polished (6um) and etched (watered acid mix) to see grains. Got stuck in holder and bent during removal process. There after kept in air filled test tube.

6-30: New stick [New Run #16, Test #1, 800C 5hr, 6/30/04] removed from N<sub>2</sub> box in N<sub>2</sub> filled test tube, polished (6um) and etched (all acid mix) to see grains.

7-7: Re-polished New Run #16...(6um) and etched (all acid mix) to see grains. There after kept in air filled test tube.

7-8: SEM images of New Run #16 (vacuum conditions). Air filled test tube.

7-12: Re-polished New Run #16 (6um) and etched (all acid) to see grains. SEM images of New Run #16 (vacuum conditions). Air filled test tube.

7-22: Measured sample dimensions. Air filled test tube.

8-6: Removed remaining two sticks [Run #16, 800C 5hr, 8/6/04] from N<sub>2</sub> box in N<sub>2</sub> filled test tubes.

Taken to SEM to image for Cris, SEM filament burnt out before images could be taken, left samples under vacuum in SEM. Samples marked with knife to indicate area imaged.

8-9: Examine Run #16...8/6/04 in SEM (vacuum conditions). Air filled test tube. Given to Cris later in the day.

### **Run #18**

Test #2

800C 2hr

4 Nb sticks

1 stick [Run #18, Test #2, 800C 2hr] placed in N<sub>2</sub> filled test tube.

6-24: Run #18... polished (6um) and etched (watered acid mix) to see grains. Air filled test tube.

6-29: Side of Run #18 polished (6um) and etch (all acid mix) to see grains. Air filled test tube.

7-2: Re-polished surface of Run #18 (6um) and etched (all acid mix) to see grains. SEM images of Run #18 (vacuum conditions). Air filled test tube.

7-8: SEM images of Run #18 (vacuum conditions). Air filled test tube.

7-12: Re-polished Run #18 (6um) and etched (all acid) to see grains. Poor polish...images useless. Air filled test tube.

7-22: Measured sample dimensions. Air filled test tube.

### **Run #19**

Test #3

800C 1hr

4 Nb sticks

7-6: 1 stick [Run #19, Test #3, 800C 1hr] polished (6um) and etched (all acid mix) to see grains. Air filled

test tube.

7-9: SEM images of Run #19 (vacuum conditions). Air filled test tube.

7-12: Re-polished Run #19 (6um) and etched (all acid) to see grains. SEM images of New Run #16 (vacuum conditions). Air filled test tube.

7-22: Measured sample dimensions. Air filled test tube.

## **Run #20**

Test #4

700C 5hr

4 Nb sticks

7-6: 1 stick [Run #20, Test #4, 700C 5hr] polished (6um) and etched (all acid mix) to see grains; a few extra drops of etchant. Air filled test tube.

7-9: SEM images of Run #20 (vacuum conditions). Air filled test tube.

7-12: SEM images of New Run #16 (vacuum conditions). Air filled test tube.

7-22: Measured sample dimensions. Air filled test tube.

8-4: New stick [700 5h, 8-4-04] removed to lightly BCP etch (20um). Air filled bag.

8-5: SEM images of 700 5h (vacuum conditions). Air filled test tube.

8-6: Removed new stick [Run #20, Test #4, 700 5hr, 8-6-04] from N<sub>2</sub> box in N<sub>2</sub> filled test tubes. Taken to SEM to image 'clean' surface, SEM filament burnt out before images could be taken, left sample under vacuum in SEM.

8-9: SEM image of Run #20...8-6-04 (vacuum conditions). Air filled test tube.

## **Run #22**

Test #5

500C 24hr, 800C 5hr, "Plateau" Sample

4 Nb sticks

7-27: 1 stick [Run #22, Test #5, 500 24, 800 5hr] polished (6um) and etched (all acid mix) to see grains. Air filled test tube.

7-28: SEM images of Run #22 (vacuum conditions). Air filled test tube.

## **Run #23**

Test #6

600C 10hr

4 Nb sticks

8-2: 1 stick [Run #23, Test #6, 600C 10hrs] polished (6um) and etched (all acid mix) to see grains. SEM images of Run #22 (vacuum conditions). Air filled test tube.

8-3: New stick [600C 10hr, 8-3-04] removed from N<sub>2</sub> box in N<sub>2</sub> filled test tube.

8-4: 600C polished (6um) and etched (all acid mix) to see grains. Air filled test tube.

8-5: SEM images of 600C (vacuum conditions). Air filled test tube.

## **Control**

No heat treatment

6 Nb sticks

6-24: 1 stick [control] polished (6um) and etched (watered acid mix) to see grains. Air filled test tube.

6-29: Side of control polished (6um) and etched (watered acid mix) to see grains. Air filled test tube.

6-30: New stick [New Control, 6/30/04] kept in nitrogen filled test tube.

7-2: Re-polished control (6um) and etch (all acid) to see grains. Air filled test tube.

7-8: SEM of New Control before polishing (vacuum conditions). Air filled test tube.

7-9: SEM images of control (vacuum conditions). Air filled test tube.

7-12: Polished New Control (6um) and etch (all acid) to see grains. SEM images of New Control (vacuum

conditions). Air filled test tube.

7-22: Measured sample dimensions.

8-3: New stick [control, 8-3-04].

8-4: control, 8-3-04 polished (6um) and etched (all acid mix) to see grains; ran out of acid mix—poor etch, and useless images. Air filled test tube.

8-5: SEM images of control (poor), 8-3-04 (vacuum conditions). Air filled test tube.

### **Cut Samples**

No heat treatment, no initial BCP etch, no initial storage in dry nitrogen

6 Nb sticks

2 sticks: used to make holder—Dave Burk still has one, or it is lost

2 sticks: polished along with Plateau and 600C samples to keep holder flat, not etched

8-4: 1 stick [10-15C 100m] BCP etched for 100 minutes with regulated temperature (not polished before).

Air filled bag.

8-4: 1 stick [Room Temp 100m] BCP etched for 100 minutes with unregulated temperature (not polished before). Air filled bag.

8-5: BCP etched samples in SEM (vacuum conditions) to see surface. Air filled bags.

8-10: 1 stick [8-10-04, BCP 100m, ~13mL acid] BCP etched for 100 minutes with regulated temperature (not polished before). Air filled bag. SEM images of 8-10-04. Air filled bag.

Other sticks in air filled bag labeled: Dan Snee 30.8.2.01.1.2, Niobium Samples.

## **Appendix B: Furnace Information**

### ***Equipment List***

- Thermcraft Furnace
  - Model #TSL-10-0-36-3C-J6063/2EA
  - 23200 W, 208 V, 65 A
  - 1200°C Maximum Temperature
  - Serial # 0166063/1A
  - FNAL ID #89560
- Thermcraft Temperature Monitor
  - Model #3-3-80-208-Y07ZP-J6063/2EA
  - 208 V, 60 Hz, 65 A
  - Serial #016063/2EA
- Echo Dry Roughing Pump
  - AEG Type AM 100 LT6 Q4
  - Model #13950
  - No. 23812299F
  - Serial #20900014075
- Pfeiffer Vacuum Turbo Pump
  - control: TCP 600 Electronic Drive Unit
  - pump: THM 1600DN 250 150-K, 2P PM P02 375
  - Serial # 12336692
- Spectra Instruments VACSCAN with Multiplier
  - Model LM6
  - 100-240 V, 50/60 Hz, 1 Amp
  - Control Unit Serial # LM6-01589005
  - Analyser Serial #LM2-89135-1D
  - RF Head Serial #LM10-01589005
  - FNAL ID #69471
- Granville Phillips 350 Ionization Gauge Controller
  - Catalog #350001
  - Serial #4957
- Granville Phillips 316 Vacuum Gauge Controller
  - Calibrated for nitrogen
  - Catalog #316001
  - Serial #2246-E

- National Instruments 24V DC Power Supply
  - Model FP-PS-4
  - Part #187999A-01
  - Serial #CECEB3
- National Instruments RS-232/RS-245 Network
  - Model FP-1000
  - Part #184120F-01
  - Serial #D0379E
- National Instruments 8 Channel Thermocouple Input
  - Model FP TB-3
  - Part #186424A-01
  - Serial #100855A
- Epson FX-85 Dot Matrix Printer
  - FNAL ID #56872

### ***LabVIEW Program Information***

A LabVIEW Virtual Instrument set up by Moyses Kuchnir collects data from the VACSCAN for monitoring purposes. This data is saved as an Excel file to allow later analysis. The LabVIEW program is set up to monitor partial pressure data and control furnace temperature. The partial pressures can be reset to accommodate other experiments. The temperatures monitored and controlled include: interior furnace zones 1, 2, and 3, the top and bottom of the furnace's exterior, the top interior baffle, above, at the center, and below the shelving unit, aluminum reference, furnace reference, and channel 7. The current partial pressures tracked are: VACSCAN total pressure, H<sub>1</sub>, H<sub>2</sub>, H<sub>2</sub>O, air, N<sub>2</sub>, and CO, O<sub>2</sub>, and CO<sub>2</sub>. The VACSCAN total pressure does not include molecules with masses of less than ten atomic mass units.

LabVIEW regulates the temperature ramp rate to 300 degrees per hour or five degrees per minute. The VI is also set to stop temperature ramping if the VACSCAN total pressure exceeds  $5 \times 10^{-8}$  Torr. The temperature ramp will begin again when the VACSCAN total pressure drops to  $2.5 \times 10^{-8}$  Torr.

### ***Additional Information***

Some capabilities of the VACSCAN apparatus include: partial pressure readings, a leak check system, and spectrum analysis of molecules up to 100 amu. The VACSCAN uses "a quadrupole mass spectrometer [as] a partial pressure measuring instrument. It can, however, be used to mimic the action of a total pressure gauge, more precisely an ion gauge. By switching the DC potential off the rods the quadrupole is effectively grossly under resolved. This allows most of the ions generated in the ion source to pass down the filter and strike the detector. Using this method, the mass spectrometer takes a true total pressure measurement rather than performing a summation of the partial pressure measurements" (Technical Note, Vacscan Manual, page 31).

The Granville Phillips Ionization Gauge measures the total pressure, including the smaller molecules, such as hydrogen. This gauge is monitored manually during the furnace bakes, but is not hooked into the LabVIEW system.

The pressure gauges are located below the bottom set of baffles. The total volume under vacuum is 81.5 liters. The dimensions of the furnace chamber available for samples are: diameter of 8 inches, height of 27 inches.

### ***Procedure Summary***

In dealing with the niobium samples for the heat treatment study furnace work at Lab 7, clean conditions are maintained as much as possible. Powderless latex gloves of CR 100 rating are worn when handling the samples. The samples are stored in nitrogen filled plastic test tubes in a dry nitrogen box in order to prevent as much oxidation as possible. Niobium wire is used to secure the samples to the shelving unit. The treated samples are stored with the wire wrapped end toward the opening of the test tube to keep track of possible contamination. The furnace is open to atmospheric conditions as little as possible between runs to keep the heating chamber free of excess particles, especially oxygen. When the furnace is open, as during sample installation, low pressure nitrogen is pumped into the furnace to keep oxygen out of the chamber. Once the samples are installed and the all metal flange at the chamber opening is secured, a purging process is undertaken with nitrogen to clean out particles that may have entered the furnace chamber. The vacuum is pumped to one Torr, and then brought back to atmospheric pressure (640 Torr) three time before the final pump down. The Echo Dry roughing pump prevents too many particles from entering the turbo, and decreases the risk of condensation during the purging process. The VACSCAN/LabVIEW interface is monitored during the ramp up to the desired temperature in order to keep outgassing plateaus to a minimum. A typical time scale is as follows:

- Day 1: open furnace, install samples
- Day 2: temperature ramp up and bake
- Day 3: cool down
- Day 4: open furnace, retrieve samples.

See Eileen Hahn for further details or technical instructions.

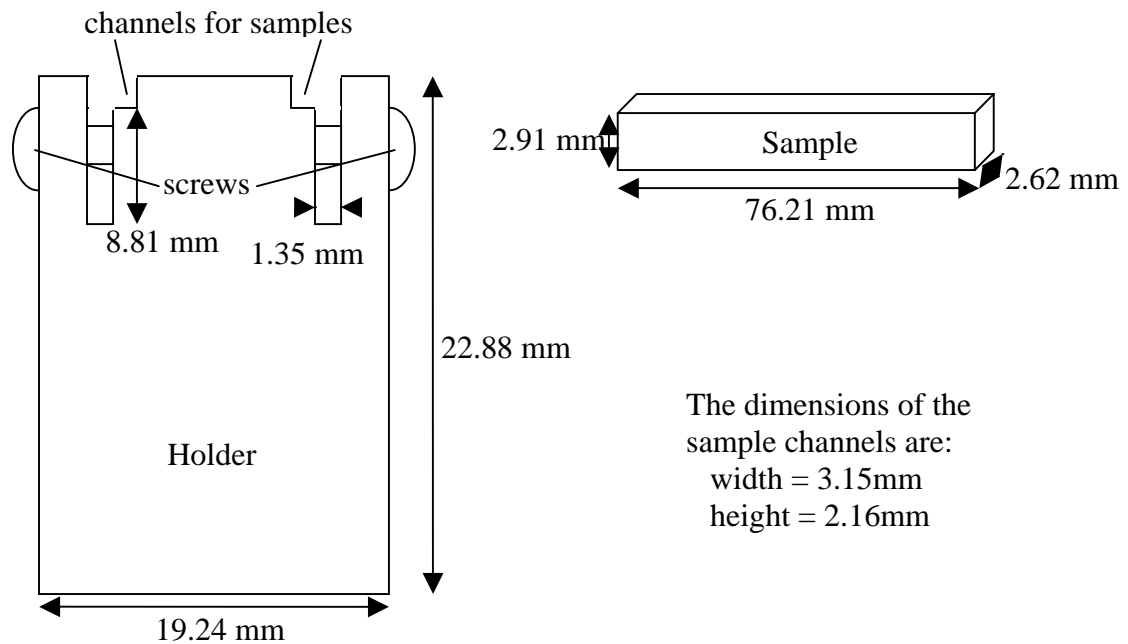
## **Appendix C: Polishing Information**

### ***Equipment List***

- Low Speed Polisher
  - FNAL ID 49976
- Leco 8” Polishing Disc, Offset for Texmet
  - Part No. 810-482
- Buehler Metadi Diamond Suspension, oil base
  - 30 micron: No. 40-6222
  - 15 micron: No. 40-6544
  - 6 micron: No. 40-6540
- Samples
  - pure niobium sticks 2.6mm x 2.92mm x 190.5mm
- Sample Holder
- Acid
  - Hydrochloric (HCl)
  - Hydrofluoric(HF)
  - Nitric (HNO<sub>3</sub>)
- Screw Driver, Putty Knife, Sharpie Pens (various colors), Dawn Dish Soap, Beaker, Test Tube, Pipette, Gloves
- Paper Towels:
  - Kimberly-Clark Professional Kaydry delicate task wipers
- QCapturePro, version 5.0.0.16
- Jasc Paint Shop Pro 8, version 8.10
- Objective Compound Microscope: MicroStar American Optical
  - FNAL ID 39018
- Stereo Compound Microscope: Zeiss Stemi SV8
  - FNAL 63964
- Vision Engineering Mantis FX
- Scanning Electron Microscope: Jeol JSM-5900LV SEM
  - Serial #MP17710042

### ***Procedure***

In order to see the grain pattern on a metal sample, the sample usually needs to be polished smooth and then etched with an acid mixture. The acid attacks the grain boundaries more aggressively than the grains themselves. But first the sample needs to be polished. For this study, polish the samples with diamond slurries of decreasing grit. A metal holder consists of essentially a block of aluminum with channels for two samples to rest in. Secure the samples by tightening the outside edge of the channel with screws. Figure 1A shows a sketch of the study samples and an end view of the holder; the dimensions noted pertain to this study—make alterations for a new holder to suit other samples. The length of the holder should, of course, be enough to accommodate the sample, in this case 80mm. Once secure, begin polishing the sample.



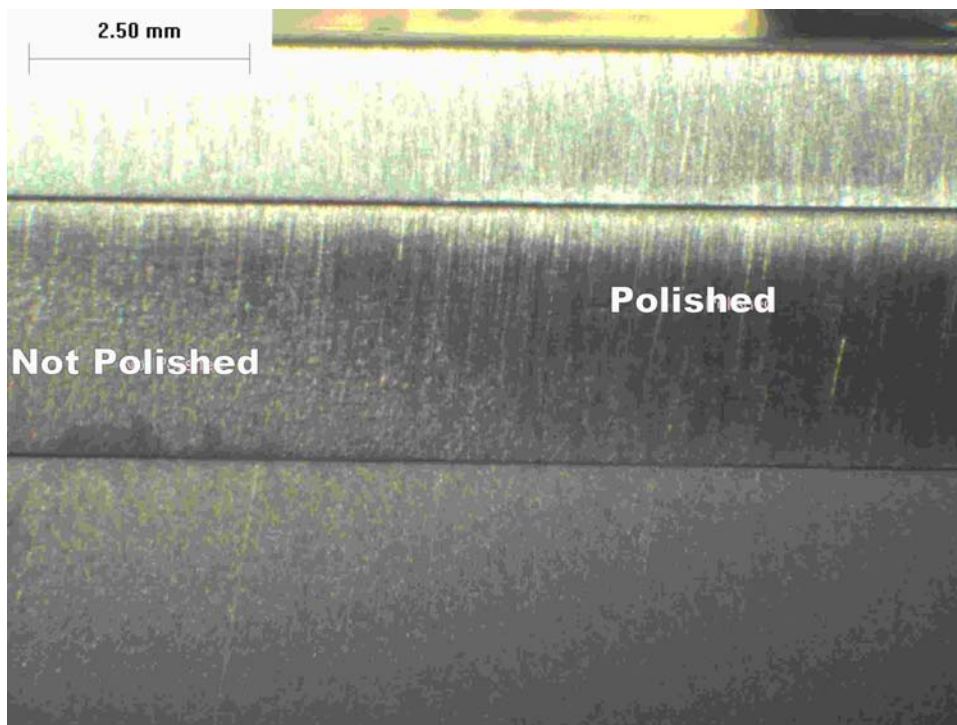
**Figure 1A: Holder and Sample**

### ***To Prepare Lathe***

1. Select a turntable.
2. If there is already a pad on the turntable, remove the pad (a putty knife is handy to get it started).
3. Thoroughly wash and dry the turntable.
4. Position and secure turntable on turning screw.
5. Place a new pad on the turntable avoiding air bubbles under the pad.
6. Spray on desired diamond slurry mixture until damp and distribute over turntable with sample.
7. The lathe is now ready to use for polishing.

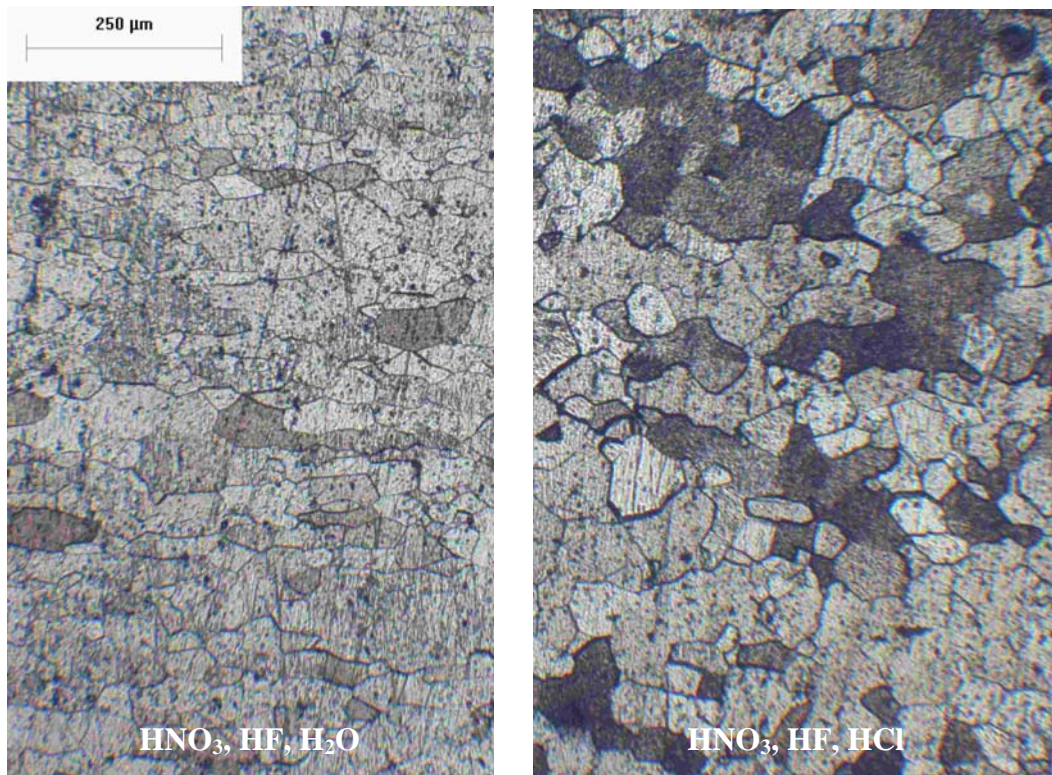
### *To Polish*

1. Secure samples in holder and be sure to mark which sample is which. In this case, variously colored Sharpie pens were used to color the end of the sample and it's holder channel.
2. Turn on polisher to lowest setting (one click to the left) with 30 micron diamond slurry wetting the pad on the surface.
3. Carefully hold sample to spinning polishing surface for one minute. The pressure on the sample should be enough to hold it in place and yet allow the sample to glide over the surface of the polisher.
4. Look at samples to determine whether the entire sample surfaces are polished. See Figure 2A for an example of a polished versus unpolished surface. Note the direction of the marks on the surface.
5. Turn samples 90° and repeat steps 3 and 4. The marks should all be in this new orientation. If they are not, continue until they are.
6. Repeat steps 3 through 5 until samples are completely polished.
7. Thoroughly wash samples in holder with soap and water.
8. Move to the next grit diamond slurry, in this case 15 microns, and repeat steps 6 and 7.
9. Move to the next grit diamond slurry, in this case 6 microns, and repeat steps 6 and 7.
10. Further grits may be used, but for the purposes of this study, no further steps were taken.



**Figure 2A:** Polished versus Not Polished

There are many combinations of acid to use for the final, short etch under the microscope, but only two acid mixtures were used during the course of the study. The first mixture, suggested by customary practice at the materials lab, was composed of one part nitric ( $\text{HNO}_3$ ), one part hydrofluoric (HF), two parts distilled water ( $\text{H}_2\text{O}$ ). This mixture brought out the grain boundaries decently well, but not to the desired level. An article<sup>4</sup> with excellent pictures of etched niobium was found that suggested a second mixture of one part nitric ( $\text{HNO}_3$ ), one part hydrofluoric (HF), two parts hydrochloric (HCl). This etch mixture brought out the grains better. Thus, the second mixture is recommended for the final, short etch process. Figure 3A shows a comparison of the effects of these etching mixtures on grain visibility.



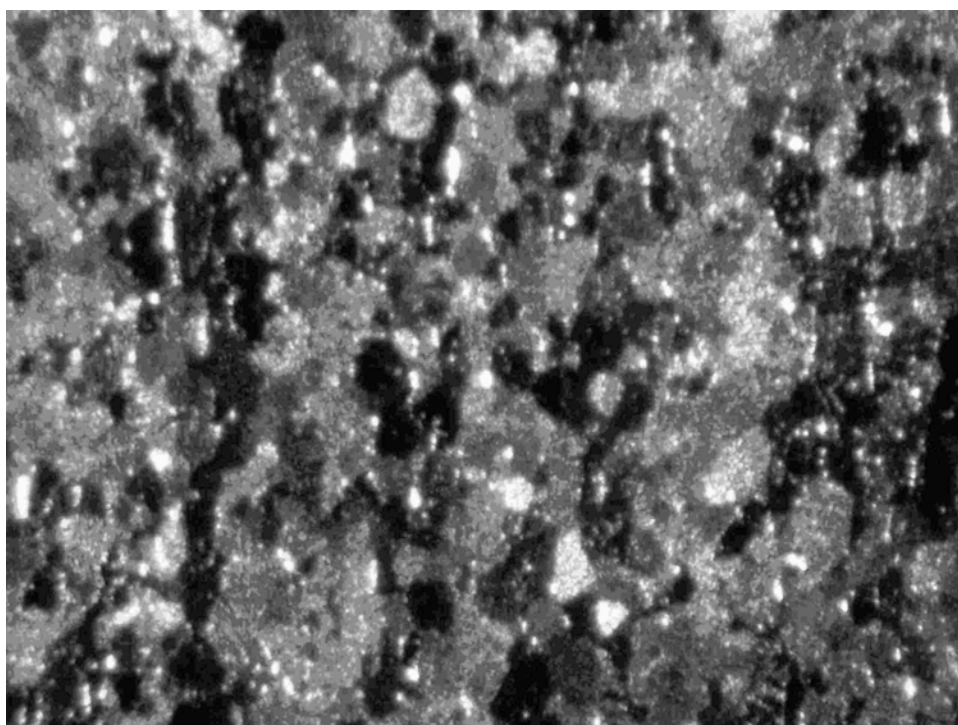
**Figure 3A:** Comparison of Acid Mix Effect on Grain Visibility

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<sup>4</sup> Jefferson F.C. Lins, Hugo R.Z. Sandim, Rosinei B. Ribeiro , and André L. Pinto. "Determination of Grain Size Distribution in Niobium Using an Image Analysis Routine." *ACTA Microscopica* 12.1 (December 2003): 121-124. SBMM. 24 June 2004 <http://www.sbmm.org.br/actar/trabalhos/24.pdf>.

### *To Etch*

1. Remove sample from holder, wipe any leftover diamond slurry from the unpolished surfaces, and place under low magnification microscope.
  2. Using a modified plastic pipette (the tip has been drawn out to allow single drops of liquid to pass), place a drop of the acid mixture on the sample.
  3. Allow air bubbles to expend themselves.
  4. Repeat steps 2 and 3 until grains are clearly visible, see Figure 4A.
  5. Rinse sample with water to remove excess acid and residue.
  6. The sample is now ready to be examined under higher magnification microscopes. For better results, this should be done within a day of etching.
- \* Appropriate gloves must be worn when working with acid!
  - \* Store test tube with acid and pipette in beaker in acid cabinet.



**Figure 4A:** Example of Etched Niobium Sample at 6.4x under Stereo Compound Microscope

A diamond slurry polish of six microns followed by an etch of nitric, hydrofluoric, and hydrochloric in a 1:1:2 acid mixture is sufficient to reveal grain boundaries. Etching pits, such as those seen on poorly electropolished materials, often occur. However, these pits typically do not interfere with grain size measurement, and thus pose little problem to this study.

## **Appendix D: Imaging and Software Help Sheet**

Every computer program has its own foibles. Discovering all these quirks yourself can take a while, so here is a brief guide to get you started.

### ***QCapture Pro***

After opening this program click on the camera icon on the tool bar. This will enable the selected camera to operate. A tool box will open either the Advanced or Basic Dialog. To select the appropriate camera get into the Advanced tool box.

#### ***Camera Selection***

1. Setup tab
2. Select Device drop down menu
3. QI Device 9259 is the camera on the Stereo Compound microscope
4. QI Device 9284 is the camera on the Objective Compound microscope
- \* In order for the Stereo Compound microscope's camera to take pictures the knob configuration should be: left out, right in.

To acquire an image select a camera as outlined above and choose the Preview button. A new window will open that displays what the camera sees. If the window is black, the exposure level is too low. If the window is white, the exposure level is too high. The image tends to be somewhat green. Change this with the white balance. The Auto White Balance and Auto Exposure functions do a fairly good job to correct such problems.

#### ***Image Adjustment***

1. Basic Dialog box
2. Click the More>> button
3. Click the Auto White Balance button
4. Click the Calculate button
5. Click the Auto Exp Area button
6. Click the Calculate button
- \* If desired, manual control of the exposure level is possible, but be sure to lock the preview exposure (ExpPvw) to the acquisition exposure (ExpAcq). The icon next to the manual exposure control sliders should show a locked lock.

The focus of the camera is supposed to be adjusted such that what is seen through the microscope lenses appears on the computer screen, but the focus can be off, particularly on the objective microscope. Turn the screen toward you and adjust the fine focus on the microscope, while watching on the computer. At the bottom of the preview window focus measurements are displayed. The best focus seems to be when the two numbers are equal and as large as possible. When the desired image adjustments are finished, click the Snap button to capture the preview window. If desired, a calibrated marker can be placed on the image.

### *Marker*

1. Click the Wrench (Spatial Calibration) icon on tool bar
  2. Select the appropriate magnification from drop down menu
  3. Click the Marker button
  4. Input the desired marker length
  5. Click the Okay button
  6. Follow instructions on screen, namely place the marker at desired position and right click to “burn” the marker on the image.
- \* The marker length may need to be adjusted by trial and error to show the entire length of the marking line.

Saving the image produces a TIFF file that opens only from the computer that took the image. Consequently, open the image in Paint Shop Pro and save again to open the file elsewhere. Saving as a JPEG reduces the file size, as does converting to 8-bit color.

### *JSM-5900 Scanning Electron Microscope*

Before using the SEM software, make sure the Temperature Stabilizer (the tan box on the other side of the room) has both pink and red lights glowing. Then turn on the main power to the SEM by switching the key on the front of the desk the SEM sits on to the ‘On’ position. Then the program may be opened.

### *Getting Started*

1. Press the Menu button to see a list of options and choose Specimen Exchange. If the SEM was under vacuum, press the Vent button. Once the Vent function is complete, there will be a beep, at which point the door may be opened.
  2. Arrange the specimen in the holder so that the two are flush at the top (for auto function purposes).
  3. Use the special specimen holder tongs to place the specimen in the chamber.
  4. Close the chamber door and press the Evac button. When this shows ‘Ready,’ the chamber is prepared for the electron beam.
  5. Press the H T button to turn on the beam.
  6. Adjust the working distance to the desired level, but no less than 10.
- \* As a precaution against too small a working distance, keep the joy stick control on X/Y when not adjusting working distance.

Pressing the ACB button at the top of the screen triggers the scanning electron microscope software’s auto contrast feature. Manual manipulation is also possible in the SCAN1 mode. Use the SCAN2 function when “previewing” the image you desire to obtain. After adjusting the image to the desired focus, contrast, spot size, etc, click the SCAN4 button.

### *Imaging Tips*

1. Higher spot sizes yield less graininess in final images.
2. Shorter working distances yield better resolution.
3. If ACB button causes image to become completely white, reduce magnification and try again.
4. When using the backscattering function, turn off the camera for image acquisition.

To “snap” an image, either click the Freeze button or the SCAN4 button. Once the image is frozen, change the marker on the image to reflect various information such as working distance, spot size, and beam setting. The preferred marker set-up is outlined below. Click File>>Save Image File to keep the image. There is a CD burner on the computer to enable transportation of image files.

*Marker*

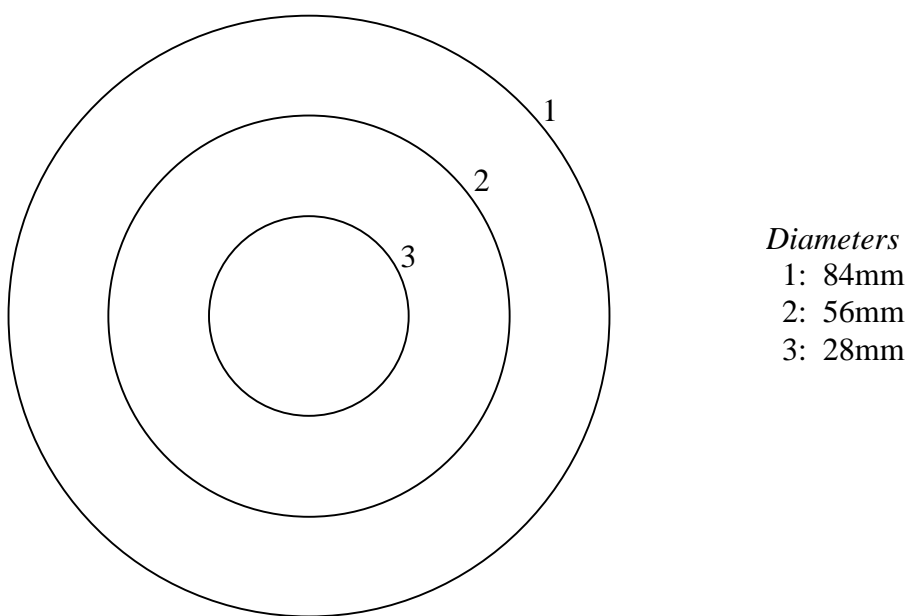
1. Select Set-up on tool bar
2. Select Photo Data on drop down menu
3. Check data to be displayed, i.e.: Acc. Volt, Micron, Magnification, Note, Background
4. Choose information to be displayed in the Note: WD/Spotsize/Signal
5. Choose the Background: Image

## **Appendix E: ASTM E 112 Intercept Method to Measure Grain Size<sup>5</sup>**

In an effort to standardize grain sizes in the materials industry, the American Society for Testing and Materials, ASTM, has outlined such measurement procedures. They desire to move away from the planimetric method as it assumes the grain geometry to be square, which is not necessarily a good approximation. The intercept method gives a better indication of grain size. This method may utilize either straight lines or circles. However, if grain elongation aspect ratio is greater than 3:1, use straight lines. Since the grain elongation aspect ratio is not greater than 1.5 for the study samples, circles will be used in this study. A brief outline of the Abrams Three-Circle Procedure follows.

### ***Intercept Method: Abrams Three-Circle Procedure***

1. Place three concentric circles (Figure 1E) with a known total line length on the desired image. This set of circles is a field. Figure 1E also lists the template circle diameters.
  2. Count the number of grain boundaries the circles intersect,  $P_i$ .
  3. Determine the number of grain boundary intersections per unit length of test line,  $P_L$  (Equation 1E).
  4. Calculate average intercept length,  $l$  (Equation 2E).
  5. Repeat for each field.
- \* The ASTM grain size,  $G$ , can also be calculated for each field (Equation 3E).



**Figure 1E: Concentric Circles**

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<sup>5</sup> “Standard Test Methods for Determining Average Grain Size (E112-96).” *1998 Annual Book of ASTM Standards* Volume 03.01: 229-251. 1998.

**Equation 1E**—Number of Grain Boundary Intersections per Unit Length of Test Line,  $P_L$

$$P_L = \frac{N_i M}{L}$$

$N_i$ : number of intersections on field (circle set)

$M$ : magnification = measured length of marker/indicated length of marker

$L$ : Total test line length, with circles above = 527.79mm

**Equation 2E**—Average Intercept Length,  $l$

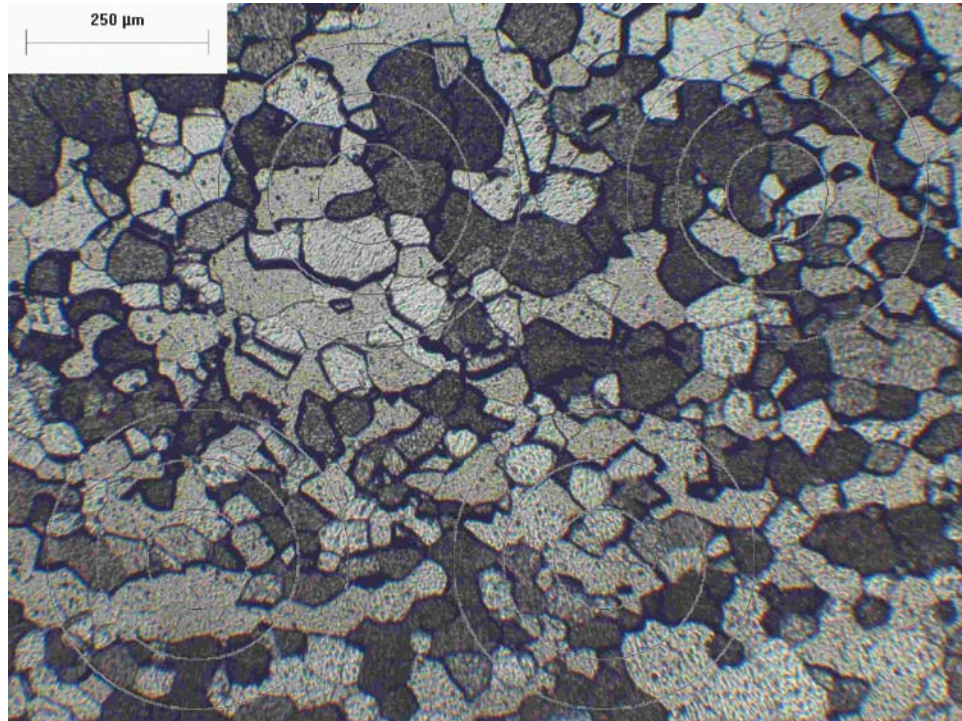
$$l = \frac{1}{P_L}$$

**Equation 3E**—ASTM Grain Size,  $G$

$$G = -6.644(\log l) - 3.288$$

$l$  in mm

The ASTM Standards recommend 400 to 500 intersection counts. “For most grain structures, a total count of 400 to 500 intercepts or intersections over 5 to 10 fields produces better than 10% relative accuracy” (pg 239). Figure 1E shows an example of concentric circle placement using a transparency of the circles laid on top of an optical microscope image.



**Figure 1E:** Concentric Circles on Optical Microscope Image of 800C, 5 hour run

Statistical analysis, as presented in Section 15 of ASTM E 112, follows. Twelve fields were used for each sample: four fields from each of three separate images of each sample. The mean value of the average intercept length,  $l_{avg}$ , is used to determine the standard deviation,  $s$  (Equations 4E and 5E). The number of fields and the standard deviation are used to calculate the uncertainty in  $l_{avg}$  (Equation 6E). The average intercept length will be used as the average grain diameter.

**Equation 4E**—Average Intercept Length,  $l_{avg}$

$$l_{avg} = \frac{\sum l_i}{n}$$

$l_i$ : average intercept length for a given field  
 $n$ : number of fields

**Equation 5E**—Standard Deviation,  $s$

$$s = \left[ \frac{\sum (l_i - l_{avg})^2}{n - 1} \right]^{1/2}$$

**Equation 6E**—Uncertainty,  $\delta$ , in  $l_{avg}$

$$\delta = \left[ \frac{\text{Average } P_L}{n} \right]^{1/2}$$

Another useful statistical measurement is the 95% confidence interval. Out of 100 measurements, 95% should fall within this limit. Equation 7E gives this value. Table 7 of ASTM E 112 procedure gives the variable  $t$  for 12 fields (pg 240). Lastly, the percent relative accuracy should be less than 10% (Equation 8E).

**Equation 7E**—95% Confidence Interval, 95% CI

$$95\% \text{ CI} = \frac{ts}{\sqrt{n}}$$

$t = 2.201$

**Equation 8E**—Percent Relative Accuracy, %RA

$$\% \text{ RA} = \frac{95\% \text{ CI}}{l_{avg}} * 100$$

## **Appendix F: Yield Stress Measurement**

Since grain size is now known, a Hall Petch plot can be used to determine yield stress. The Hall Petch equation is in the form of a linear function (Equation 1F) represented by the straight line on the Hall Petch plot. Equation 2F uses  $x_1=.05$ ,  $y_1=45$ ,  $x_2=.15$  and  $y_2=73$  to determine slope, Equation 3F yields the y-intercept, and Equation 4F gives the yield stress as determined by grain size.

### **Equation 1F—Hall Petch Equation**

$$\sigma_y = \sigma_0 + kd^{-1/2}$$

$\sigma_y$ : yield stress

$\sigma_0$ : intrinsic yield stress

$k$ : constant for given material

$d$ : grain size

### **Equation 2F—Slope, $m$**

$$m = \frac{y_2 - y_1}{x_2 - x_1} = 280$$

### **Equation 3F—Y-Intercept, $b$**

$$b = y_1 - mx_1 = 31$$

### **Equation 4F—Yield Stress, $\sigma_y$**

$$\sigma_y = 31 + \frac{280}{\sqrt{d}}$$

$\sigma_y$ : yield stress in MPa

$d$ : grain size in  $\mu\text{m}$

Stress is a function of load per cross sectional area, and allowable stress is a function of failure stress and safety factor. In this case, yielding could be considered failure. Thus, the allowable load at any point on the cavity may be determined for a chosen safety factor and known cross sectional area (Equation 5F)<sup>6</sup>. This simple equation can lead to quite a complex analysis of a structure depending on its geometry. Therefore, the only allowable load calculated here will be for the iris, where there is the least cross-sectional area and smallest allowable load.

### **Equation 5F—Allowable Load, $P_{allowable}$**

$$P_{allowable} = \frac{\sigma_y A_{cs}}{SF}$$

$\sigma_y$ : yield stress in MPa

$A_{cs}$ : cross sectional area in  $\text{m}^2$  at point where load is applied =  $2.23 \times 10^{-4}$

$SF$ : safety factor = 2

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<sup>6</sup> Norman E. Dowling. *Mechanical Behavior of Materials: Engineering Methods for Deformation, Fracture, and Fatigue, 2<sup>nd</sup> Edition*. New Jersey: Prentice Hall, 1999.